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EVALUATION OF THE LEWISITE PROTECTION PROVIDED BY THE OQUIRRH MOUNTAIN FACILITY (OMF) PROCESS FILTER

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14. ABSTRACT The Program Manager, Elimination of Chemical Weapons, tasked the U.S. Army Edgewood Chemical Biological Center (ECBC), Chemical Biological Filtration Team, to estimate the protection time afforded by the Oquirrh Mountain Facility's (OMF) process filter for lewisite. To accomplish this, it was necessary to construct a test apparatus to measure lewisite breakthrough data and develop a defensible methodology to convert these data into a process filter performance estimate. This report provides all the details of the experimental system design, data measured, and analysis used to generate the process filter protection time. In addition, data were measured for two additional adsorbents: (1) 12 x 30 mesh ASZM-TEDA carbon to establish protection times for a C2 canister, and (2) 6 x 16 mesh ASZM-TEDA carbon to determine if using this material would be more suitable than using the Vapure 612 in the process filter. The process filter using the Vapure 612 carbon currently installed at the OMF site will prevent lewisite from penetrating the first 3-ft bed section for at least 5 years. This estimate assumes the worst operating conditions possible. There is a potential for false positives due primarily to the fact that the Vapure 612 will not retain HCl. It is recommended to change one filter bank of Vapure 612 carbon with the Calgon's ASZM-TEDA carbon to prevent this.

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PREFACE

The work described in this report was authorized under MIPR No. 3BICR 307DA. This work was started in November 2002 and completed in May 2003.

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EVALUATION OF THE LEWISITE PROTECTION PROVIDED BY THE OQUIRRH MOUNTAIN FACILITY (OMF) PROCESS FILTER

1. INTRODUCTION

The U.S. Army Chemical Materials Agency (CMA), Aberdeen Proving Ground, MD, is responsible for the demilitarization and disposal of chemical agents and munitions. Lewisite [2-Chlorovinyldichloroarsine; chemical form: ($C_2H_2AsCl_3$)] is a vesicant agent that contains arsenic that requires chemical neutralization rather than incineration. The Oquirrh Mountain Facility (OMF), formally named Chemical Agent Munitions Disposal System (CAMDS), has been tasked with neutralizing the stockpile of ton containers (TCs) and 10 empty TCs of lewisite stored at the facility.[1] As a vesicant agent, lewisite is considered a suspected carcinogen. Exposure to lewisite causes intense pain on contact. Exposure to the eyes, if not decontaminated immediately, will result in either permanent injury or possible blindness within 1 min of exposure. When inhaled in high concentrations, it may be fatal within 10 min. This agent can also cause sensitization and chronic lung impairment.

Lewisite is difficult to detect because it is reactive, hydrolyzes readily, and decomposes upon heating. The following chemical reaction combines the reagent 1,2-Ethanedithiol [EDT; chemical form: $HSCH_2CH_2SH$] with lewisite to form a lewisite derivative (LD) and hydrochloric acid (HCl):

$$ClCH=CH\text{-}As\text{-}Cl_2 + HSCH_2CH_2SH \rightarrow \underbrace{ClCH=CH\text{-}As\text{-}SCH_2CH_2S}_{(LD)} + 2\,HCl$$

LD is not very reactive and is thermally stable (does not decompose on heating), which allows it to be transported through heated sample lines to the air monitoring equipment.

The Program Manager, CMA Elimination of Chemical Weapons (PM/ECW) Group tasked the Chemical Biological Filtration Team, U.S. Army Edgewood Chemical Biological Center (ECBC), to estimate the protection time afforded by the OMF process filter for lewisite. To accomplish this, it was necessary to construct a test apparatus to measure lewisite breakthrough data and develop a defensible methodology to convert these data into a process filter performance estimate. This report provides all the details of the experimental system design, data measured, and analysis used to generate the process filter protection time. In addition, data were measured for 12 x 30 mesh Carbon-Activated, Impregnated, Copper-Silver-Zinc-Molybdenum-Trietheylenediamine (ASZM-TEDA) carbon to establish protection times for a C2 canister; and 6 x 16 mesh ASZM-TEDA carbon to determine if using this material would

[1] Bruce, B.; Mitchell, A.R. *Lewisite Neutralization System Monitoring Plan;* Site Plan 91-03; Deseret Chemical Depot: Stockton, UT, 2003

be more suitable than using the NORIT® Americus, Incorporated (Atlanta, GA) Vapure 612 in the process filter.

2. OBJECTIVE

The objective of this study was to determine the protection time afforded by the process carbon filter against lewisite.

3. KEY CONCLUSIONS

The key conclusions resulting from this study follow:

a. The process filter using the Vapure 612 carbon currently installed at the OMF site will prevent lewisite from penetrating the first 3-ft bed section for at least 5 years. This estimate assumes the worst operating conditions possible.

b. Hydrochloric acid (HCl) vapor will not be retained on the Vapure 612 carbon bed of the process filter.

c. The potential for a lewisite false positive detection is likely to occur in the process-filter MINICAMS monitoring equipment. (A detailed discussion is provided in Section 5.2). This detection will not occur because of lewisite breakthrough, but will result from the reaction between the eluting HCl process vapor and any chlorovinylarsenic oxide contamination formed in the detector's heated sampling line either from prior lewisite calibration injections or vapor exposure. Although the reaction mechanism of the HCl/chlorovinylarsenic oxide was not validated in this study, it has been reported in the literature.[2]

d. To prevent false alarms, it is recommended that a basic form of impregnated activated carbon, one that has a high capacity for acid gas removal, e.g., Calgon's ASZM-TEDA carbon, be used to prevent the elution of HCl gas from the filter effluent stream, thus eliminating the formation of lewisite in the sampling lines. The ASZM-TEDA is currently used in military personnel gas masks and U.S. Army research and development chemical surety laboratories. This carbon is available in the same particle size (6 x 16 mesh) as the Vapure 612, and no hardware changes are needed. Only one bank of filters needs to have the ASZM-TEDA carbon.

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[2] Clark, D.N. *Review of Reactions of Chemical Agents in Water*; Task No. 80; Battelle Columbus Division: Columbus, OH, 1989 UNCLASSIFIED Report (AD-A213 287).

4. APPROACH

First, consider the process carbon-bed dimension and the typical and extreme operating conditions that the filter may experience. Table 1 provides a summary of these parameters.

It is not practical to measure the breakthrough times for a 3-ft deep, 4-ft diameter bed under the feed conditions given in Table 1. A more practical approach is to measure data using much shallower beds that will produce breakthrough times on the order of hours or a few days. Small diameter tubes are used (4.1 cm i.d.). The feed flow rate to the small tube is set to give the exact feed velocity of the process filter at the highest possible operating temperature (12.0 cm/s). Using different bed depths, one can construct a life thickness curve by plotting breakthrough time on the y-axis versus bed depth on the x-axis. The point where the curve intersects the x-axis is the critical bed depth. The slope of the life thickness curve is then proportional to the adsorption capacity of the adsorbent and is independent of mass transfer rate processes and wall effects (if there are any). This value can be used to estimate the filter life since it is anticipated that the critical bed depth for these experiments will be around 2.5 cm. (Indeed, that is the case for the Vapure 612 material). Therefore, for a 3-ft bed depth, more than 97 % (35 in./36 in.) of the bed will be at capacity when lewisite breakthrough occurs.

Table 1. Process Filter Sizes and Operating Conditions.

Parameter	Value
Carbon	Vapure 612, 6 x 16 mesh
Primary section bed depth	3 ft (91.4 cm)
Second section bed	1 ft (30.5 cm)
Third section bed depth	1 ft (30.5 cm)
Total filter depth	5 ft (152.4 cm)
Diameter	4 ft (121.9 cm)
Maximum design temperature	165 °F (73 °C)
Expected operating temperature	70-130 °F (21 –54 °C)
Estimated feed concentration (average)	***6.6 mg/m^3 at 165 °F***
Design feed flow rate (at 165 °F)	700 ft^3/min (28.3 cm/s superficial velocity)
Expected feed flow rate (at 165 °F)	184 ft^3/min (7.4 cm/s superficial velocity)
Safe sided feed flow rate (at 165 °F)	***300 ft^3/min (12.0 cm/s superficial velocity)***

5. RESULTS

All the experiments completed as of 16 April 2003 are summarized in Table 2. Breakthrough data for each of these experiments is shown in Appendix A. Table 2 has three feed concentrations listed. Tubes 1 and 2 were determined the same way. The tube and the carbon mass are weighed prior to inserting the beds into the system. At the end of the experiments, the

tube and carbon are weighed again. The weight gain is the total mass fed to the bed. Since the concentration at breakthrough is so low, for practical purposes, all the lewisite that enters the bed is retained by the adsorbent. Knowing the breakthrough time and the total volumetric flow rate to each bed along with the lewisite mass gain, one can easily calculate the feed concentration. This approach can only be used in the dry experiments since it is not possible to keep a material balance on the adsorbed material. The third feed concentration in Table 2 is obtained by measuring the weight loss in the saturator. For this feed calculation, one must first calculate the mass flow rate of lewisite by taking the saturator mass loss and dividing it by the saturator's time on stream. Using the mass flow rate of lewisite divided by the total flow rate of gas to the feed manifold gives the feed concentration. These methods were used to determine the feed concentration since directly measuring the feed using standard gas chromatographic techniques proved to be impossible.

Table 2. List of All Experiments Completed as of 16 April 2003.

Date	Carbon	Feed Conc. Tube 1	Feed Conc. Tube 2	Feed Conc. (saturator)	Feed Flow Rate	Feed Temp.	Feed RH	Bed Depth
		(mg/m3)	(mg/m3)	(mg/m3)	SLPM	(°C)	(%)	(cm)
1/28/03	Vapure 612	$9.4x10^2$	$9.1x10^2$	NA	7.5	73	dry	3.0
1/29/03	Vapure 612	$9.5x10^2$	$1.0x10^3$	NA	7.5	73	dry	3.5
1/31/03	Vapure 612	$1.1x10^3$	$1.1x10^3$	$1.1x10^3$	7.5	73	dry	4.0
2/5/03	Vapure 612	$1.1x10^3$	$1.1x10^3$	$1.1x10^3$	7.5	73	dry	2.5
2/6/03	Vapure 612	$1.0x10^3$	$1.0x10^3$	NA	7.5	73	dry	4.0
2/13/03	Vapure 612	$1.6x10^3$	$1.6x10^3$	$1.7x10^3$	7.5	73	dry	3.0
2/14/03	Vapure 612	76	75	78	7.5	73	dry	3.0
2/20/03	Vapure 612	65	65	66	7.5	73	dry	2.5
2/25/03	ASZM-T 12x30	$2.5 x10^2$	$2.4x10^2$	$2.5x10^2$	6	30	dry	2.0
2/28/03	Vapure 612	67	68	68	7.5	45	dry	2.5
3/5/03	ASZM-T 12x30	$5.2 x10^2$	$5.3x10^2$	$5.4x10^2$	6	30	dry	2.0
3/13/03	Vapure 612	N/A	N/A	52	7.5	45	25	2.5
3/24/03	ASZM-T 12x30	N/A	N/A	$2.8x10^2$	6	30	40	2.0
3/28/03	ASZM-T 12x30	N/A	N/A	$2.8x10^2$	6	30	40	2.0
4/1/03	Vapure 612	N/A	N/A	59	7.5	45	25	5.0
4/11/03	ASZM-T 6x16	Instant	Break	$2.3x10^3$	7.5	73	dry	4.0
4/14/03	ASZM-T 6x16	N/A	N/A	$1.9x10^2$	7.5	45	25	4.0
4/16/03	ASZM-T 6x16	N/A	N/A	$1.7x10^2$	7.5	45	25	4.0

The experiments above are separated into three different groups based on the adsorbent tested. The main focus of this effort is to determine the protection time afforded by the Vapure 612 material. Table 3 lists the results for the Vapure 612 material currently used in the process filter. Breakthrough times in Table 3 are for the appearance of 1 TWA, 4 TWA, and

7 TWA in the effluent. All breakthrough times have been normalized to 1,000 mg/m^3 for experiments either up to 14 February 2003 or to 66 mg/m^3 for the last two experiments in Table 3.

Breakthrough times are extrapolations made from the data sets shown in Appendix A. Examine graphed results for the first eight Vapure 612 experiments shown in Figures A-1 through A-16. There are several consistent features to all these data. First, breakthrough data are noisy, and in most cases, it is difficult to accurately estimate breakthrough time. That is one reason the TWA data shown in Table 3 is so scattered. Second, noisy data is worse closer to the breakthrough value.

As mentioned earlier, two very important conclusions can be drawn from these data.

a. The process filter using the Vapure 612 carbon currently installed will prevent lewisite from penetrating the first 3-ft bed section for at least 5 years. This estimate assumes the worst operating conditions possible.

b. The potential for false positives is present. The mechanism is not validated; but, it appears that a reversible lewisite reaction could occur when HCl is present in the effluent stream.

Table 3. Estimated Breakthrough Times (min) at Three TWA Values for Vapure 612 Carbon.

		Tube 1			Tube 2		
Date	**Carbon**	**1 TWA**	**4 TWA**	**7 TWA**	**1 TWA**	**4 TWA**	**7 TWA**
1/28/2003	Vapure 612	$3.2x10^2$	$4.0x10^2$	$4.7x10^2$	$3.0x10^2$	$3.5x10^2$	$3.8x10^2$
1/29/2003	Vapure 612	$5.3x10^2$	$6.2x10^2$	$6.7x10^2$	$3.6x10^2$	$4.3x10^2$	$4.6x10^2$
1/31/2003	Vapure 612	N/A	N/A	N/A	$8.2x10^2$	$1.0x10^2$	$1.1x10^3$
2/5/2003	Vapure 612	$2x10^1$	$4x10^1$	$5x10^1$	$6x10^1$	$1.0x10^2$	$1.2x10^2$
2/6/2003	Vapure 612	$5.3x10^2$	N/A	N/A	$4.4x10^2$	$5.5x10^2$	N/A
2/13/2003	Vapure 612	N/A	N/A	N/A	$1.5x10^2$	$2.8x10^2$	$3.4x10^2$
2/14/2003	Vapure 612	$4.0x10^3$	N/A	N/A	$5.0x10^3$	N/A	N/A
2/20/2003	Vapure 612	$1.6x10^3$	$3.7x10^3$	$4.5x10^3$	$3.7x10^2$	$1.8x10^3$	$2.7x10^3$
2/28/2003	Vapure 612	$2.3x10^3$	$3.8x10^3$	$4.8x10^3$	$2.1x10^3$	$3.4x10^3$	$4.6x10^3$
3/13/2003	Vapure 612	N/A	N/A	N/A	N/A	N/A	N/A
4/1/2003	Vapure 612	N/A	N/A	N/A	N/A	N/A	N/A

5.1 Estimated Process Filter Protection Time (Dry).

Figure 1 shows a life thickness curve generated from tube test data. These represent a conservative estimate of the breakthrough times to be safe-sided.

Using the "best fit" line from this graph, we can estimate the lewisite protection time afforded by the first section of the process filter (91-cm deep) under dry conditions at a challenge concentration of 1.1×10^3 mg/m^3. In Figure 1, the slope of the line (270) is the capacity of the Vapure 612 adsorbent at the challenge concentration expressed as minutes of protection per centimeter of bed depth that can be used to calculate protection time for the filter as follows:

Estimated protection time at 1.1×10^3 mg/m^3 feed concentration (dry, 73 °C)

$270*91 - 625 = 2.4 \times 10^4$ min = 17 days

Since the actual feed concentration to the filter is more than 2 orders of magnitude lower (6.6 mg/m^3), we must modify the 17-day estimate to account for the lower feed concentration. Using data from the low concentration experiment shown in Table 3, we can safely assume that the adsorption capacity of lewisite at 6.6 mg/m^3 is at least 70% of the loading at 1,080 mg/m^3. Using the actual feed concentration and a reduction of 30% in lewisite loading due to the lower feed concentration, the breakthrough time estimate for the 6.6 mg/m^3 feed concentration at 73 °C and dry conditions can be generated as follows:

Estimated protection time at 6.6 mg/m^3 feed concentration (dry, 73 °C)

$270*0.70*91*1.1 \times 10^3/6.6 - 625 = 2.9 \times 10^6$ min = 2.0×10^3 days

This means that the filter would have to be exposed to 6.6 mg/m^3 feed CONTINUOUSLY for more than 5 years at 73 °C to produce a 1 TWA concentration in the effluent of the first 3-ft filter. This is safe-sided because data from both humid and lower temperature experiments using Vapure 612 show that the lewisite protection time is increased.

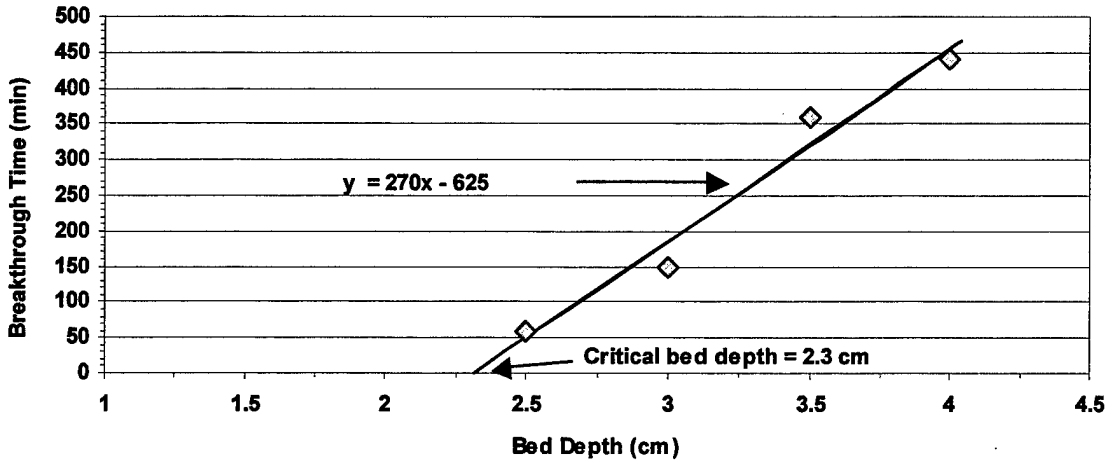


Figure 1. Life Thickness Curve for Vapure 612 at High Feed Concentration, 73 °C and Dry Conditions.

5.2 Noise Problem and Potential False Positives.

A review of the data in Figures A.17 through A.29 shows that a false positive can occur using the Vapure 612. Start by comparing the noise seen in the effluent signal using the Vapure 612 versus that seen in the four ASZM-TEDA 12 x 30 mesh experiments shown in Table 4. The results for the ASZM-TEDA besides being much less noisy are more reproducible. The following mechanism is proposed to explain these data. When lewisite reacts on the Vapure 612, it generates HCl. It is well-known that microporous activated carbon can catalyze hydrolysis reactions with acid-forming vapors such as lewisite by bringing them into close contact in the adsorbed phase. Since the Vapure 612 does not have the metal impregnates needed to retain HCl; it quickly breaks through into the effluent. Downstream of the filter bed, chlorovinylarsenic oxides that may have been deposited from previous experiments are then reversibly converted back to lewisite. We know from visual inspection that a white powder formed in the lines downstream of our test beds. The detector located downstream of this material can than respond to this small amount of lewisite generated in this reverse reaction. The data for the 12 x 30 mesh ASZM-TEDA is much less noisy because the impregnated metal salts used in producting the ASZM-TEDA have a high capacity for acid gas removal, and thus preclude the lewisite reformation. We are not saying that this occurs in the process filters, since lewisite almost assuredly has not broken through into the product line. However, it is reasonable to assume that since the Vapure 612 has little capacity for HCl, that HCl can penetrate into the product. However, one may easily speculate that after many calibrations using lewisite, if enough chlorovinylarsenic oxide is generated, then HCl from the process filter could reform the lewisite through its backwards reaction in the sample line and set off a false positive alarm. It only takes 2 or 3 ng (1.0×10^{-9} g) of lewisite to set the alarm off. This is why we recommend using the ASZM-TEDA carbon.

Table 4. Estimated Breakthrough Times (min) at Three TWA Values for ASZM-TEDA 12 x 30 Mesh Carbon.

Date	Carbon	Tube 1			Tube 2		
		1 TWA	4 TWA	7 TWA	1 TWA	4 TWA	7 TWA
2/25/2003	12x30 ASZM-T	$2.1x10^3$	$2.3 x10^3$	$2.4 x10^3$	$1.7 x10^3$	$1.8 x10^3$	$1.9 x10^3$
3/5/2003	12x30 ASZM-T	$1.1 x10^3$	$1.2 x10^3$	$1.2x10^3$	$1.2 x10^3$	$1.2 x10^3$	$1.3 x10^3$
3/24/2003	12x30 ASZM-T	$1.5 x10^3$	$1.6 x10^3$	$1.7 x10^3$	$9.8 x10^2$	$1.1 x10^3$	$1.2 x10^3$
3/28/2003	12x30 ASZM-T	$1.4 x10^3$	$1.6 x10^3$	$1.6 x10^3$	$1.1x10^3$	$1.3 x10^3$	$1.4 x10^3$

In an effort to prevent false positives, larger particle ASZM-TEDA carbon was obtained and tested. Table 5 shows the preliminary results from the large particle ASZM-TEDA. Using the 4 cm bed depth, immediate breakthrough is observed for the first test. Upon further investigation, we discovered that the particles are much larger than the Vapure 612 particles. At 73 °C, for these large particles, the critical bed depth is >4 cm. At lower feed temperatures (the next two experiments), the critical bed is <4 cm; therefore, measurable breakthrough times were obtained. However, the critical bed is still close to 4 cm, as evidenced by the noise in the breakthrough data (Figures A.30 through A.34).

Table 5. Estimated Breakthrough Times (min) at Three TWA Values for ASZM-TEDA 6 x 16 Mesh Carbon.

Date	Carbon	Tube 1			Tube 2		
		1 TWA	4 TWA	7 TWA	1 TWA	4 TWA	7 TWA
4/11/2003	6x16 ASZM-T	<15	N/A	N/A	<15	N/A	N/A
4/14/2003	6x16 ASZM-T	580	740	870	545	730	870
4/16/2003	6x16 ASZM-T	650	860	990	570	805	940

6. EXPERIMENTAL TEST SYSTEM AND PROCEDURE

6.1 Apparatus.

Figure 2 shows a schematic of the apparatus used in this work. All of the air inlet lines are located on the left-hand side of the schematic. The shaded areas indicate the components that are contained within an insulated polycarbonate box with a slight negative pressure. Polytetrafluoroethylene (PTFE) flow lines carry chemicals in this system to minimize any interaction with lewisite. The flow controller for the water saturator is located at the top on the left. This controller was used only in later experiments where humidified feed streams were used. The water saturator line is 1/4 in. PTFE tubing up where it intersects with the diluent line. The flow controller directly below the water saturator controller is the diluent flow controller.

The diluent line is 3/8 in. copper tubing. The airflow controller for the chemical saturator is below this line. The chemical saturator line is 1/4 in. PTFE tubing up to where it intersects with the diluent line. After that, the line is 3/8 in. PTFE tubing to the manifold and exiting the manifold to vent. The chemical saturator is equipped with a 4-way valve to safely isolate the chemical when there is no chemical demand. The temperature of the chemical saturator determines the vapor pressure of the chemical (lewisite). The flow rate through the saturator along with the lewisite vapor pressure fixes the mass flow of lewisite to the manifold. The sum of all three flow rates is the total airflow through the heat exchanger and subsequently to the feed manifold. The concentration of lewisite flowing to the manifold can then be calculated by dividing the mass flow rate of lewisite out of the saturator by the total flow rate of air. The purge air controller to the left and slightly below the chemical saturator meters the clean air to the bottom manifold. The bottom two controllers meter the purge gas to the effluent side of the system. Since such low lewisite concentrations (1 TWA = 0.003 mg/m^3) are being measured, it is imperative that the analytical lines are clean prior to start of each experiment. The effluent purge lines are 1/4 in. PTFE tubing.

Within the polycarbonate box, the temperature-controlled water saturator is at the top left. The saturator is equipped with a heater to maintain a constant temperature as water is evaporated. The heat exchangers used to equilibrate the diluent and chemical flow streams to the box temperature are located below the water saturator. The diluent heat exchanger is 3/8 in. copper tubing, and the chemical heat exchanger is coils of 3/8 in. PTFE tubing. The box temperature is the experimental test temperature. Two manifolds are located to the right of the heat exchangers. The top manifold is for the feed air stream, and the bottom manifold is for the clean purge air. The two are separated to prevent low-level off gassing from contaminated lines during the purge steps in the procedure. Two adsorbent beds are below the manifolds. Each bed can accept feed gas from either the feed or the purge manifold. The beds are 4.1 cm (i.d.) glass tubes with metal inserts at the top and bottom. Viton O-ring material is used to make the seals for these inserts. Each of the inserts has a 3/8 in. Swagelok fitting to connect it with the inlet and outlet lines. Thermocouples are placed at each bed outlet (bottom of bed) to identify any temperature excursions that result from chemical reactions within the bed. The dew point analyzer and a thermocouple that is inserted into the box to monitor the box temperature are located outside the box at the top.

The M48 filter that cleans the excess feed gas from the manifold is located at the top right of the schematic. This line is 3/8 in. PTFE. The 3/8 in. PTFE vent line from the purge manifold is below this line. The bed effluent flow control system (an in-line filter, a mass flow controller, a solenoid shutoff valve, and a vacuum source) continues down from the purge manifold vent to the hood. Lines exiting the bed are 1/4 in. PTFE, and they are connected to 1.5 in. diameter, 9 in. deep, and in-line filters filled with ASZM-TEDA carbon. These filters are used to remove any acid gases and breakthrough concentrations of lewisite that could potentially damage the mass flow controllers. The mass flow controllers are used to meter the challenge flow rate to each bed. A positive shut-off valve is placed between each mass flow controller and the vacuum source to stop flow to a given bed after breakthrough of that bed has occurred.

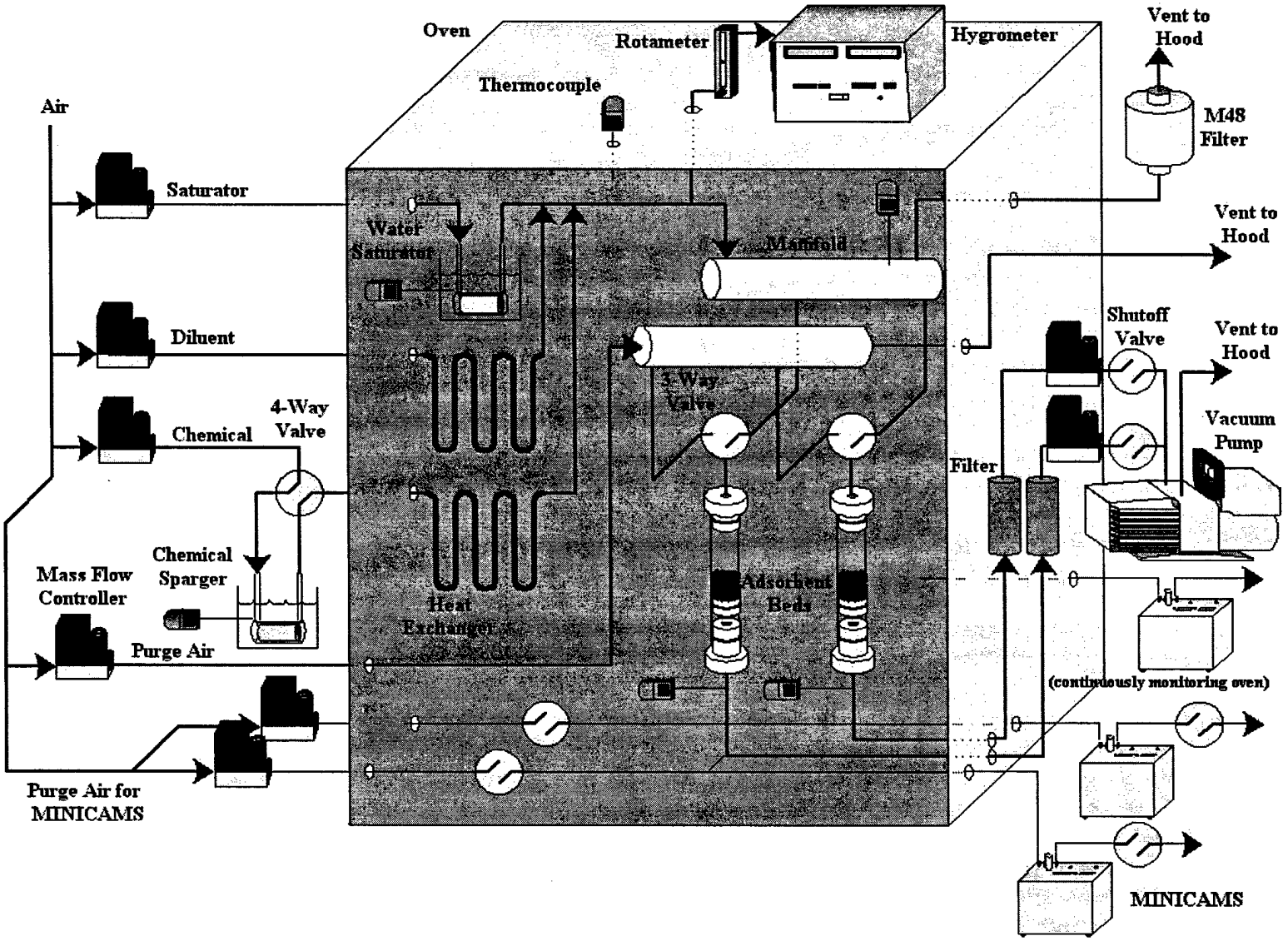


Figure 2. Schematic of Apparatus.

The three MINICAMS analyzers are at the bottom right of the schematic. The top analyzer is used as a first-entry safety monitor to monitor for lewisite concentrations that may have leaked into the box. The bottom two analyzers are used to measure effluent lewisite concentration from each bed. A 15-ft heated, 1/4 in. PTFE sample line connects each analyzer to the filter effluent near the outlet thermocouple at the bass of each filter. The 1/8 in. PTFE supply lines that direct the derivatizing reagent gas ethanedithiol (15 sccm, 200 ppm EDT in nitrogen) to each of the three lewisite monitors are not shown in the schematic. The EDT is introduced into the sample line of each analyzer near the intersection of the sample line and the bed effluent line.

6.2 Procedures.

6.2.1 Bed Filling and Installation.

- Dry carbon overnight in oven at 100 °C.
- Weigh out the appropriate amount of carbon for the desired bed depth. This value is easily determined empirically.
- Use a 1-m drop tube to fill beds.
- Weigh the filter tube assembly.
- Ensure top of bed is level with tube, use a bed leveler if required.
- Carefully place the tube inside the box.
- Disconnect water saturator from the system for a dry experiment.
- Close the box and proceed to the experimental setup.

6.2.2 Experimental Setup.

Prepare the experimental setup in the following manner:

- Set the box temperature.
- Set the chemical saturator temperature.
- Set the desired set point for each mass flow controller:
  - Water saturator (if used)
  - Diluent
  - Chemical (chemical saturator 4-way valve always defaults to the isolated position)

- Purge air to manifold
- Purge air to each analyzer and bed
- Bed challenge flow rates
- Sample line flow rate (not shown in the diagram)
- EDT flow rate (not shown in the diagram)

- Set the default start-up valve positions.

  - Bed challenge 3-way valves are set to select the purge manifold
  - Solenoids for purge air to the analyzers are closed
  - Solenoids for the bed challenge (between the mass flow controller and the vacuum source) are open
  - Solenoids for the EDT and MINICAMS effluent to vacuum are open
  - Chemical saturator 4-way valve is in the isolate saturator position

For humidified experiments, the purge manifold is not used. The equilibration period prior to the start of the feed includes the time required to equilibrate the carbon with the desired challenge RH. The chemical is isolated during this step. The feed manifold is selected using the 3-way valves above the beds. After the beds are equilibrated (usually about 3-4 hr), the 4-way valve to the saturator is switched to begin the chemical flow to the feed manifold. There is no chemical equilibration time for the humid experiments.

6.2.3 Initialization of the Control Program.

The following input is required to initialize the Control Program

- Tubes to be tested (Tube#1, Tube #2, or both).

- Chemical feed equilibration time (time period prior to bed challenge start).

- Maximum experimental run time.

- Tube shutdown criteria, which consists of two TWA values, a threshold value, and an immediate shutdown value. Two consecutive readings above the threshold TWA value are required to terminate the feed flow through a given tube. Any TWA reading at or above the immediate shutdown value obviously aborts the feed to that tube to prevent gross contamination of the sampling line. The decision to terminate the chemical challenge to a given tube results in closure of the sample tube vacuum control solenoid followed by opening of the sampling line clean-air purge solenoid valve. The injection of this clean air in front of the heated sampling line in a slight excess to the sample-line flow requirements of the MINICAMS allows for the normal operation of the

analyzer and for sample-line cleanup. The excess purge air is then safely vented through the bed and exhausts back into the feed manifold and into the vent filter.

6.2.4 End of Experiment.

At the experiment's end, perform the following steps to shut down the experiment.

- Turn off the box temperature controller.
- Turn off all flows except the analyzer purge flow lines, the EDT, and the sample flow line to the analyzers.
- When the box temperature is near room temperature, remove the beds and weigh the filter tube assembly.
- Dispose of the adsorbent properly, and carefully clean the beds to prepare for the next experiment.

Blank

APPENDIX A

LEWISITE BREAKTHROUGH DATA

Figure A.1. Breakthrough Data for the 01/28/03 Experiment
Vapure 612, 3cm, 73°C, 12cm/s, Dry

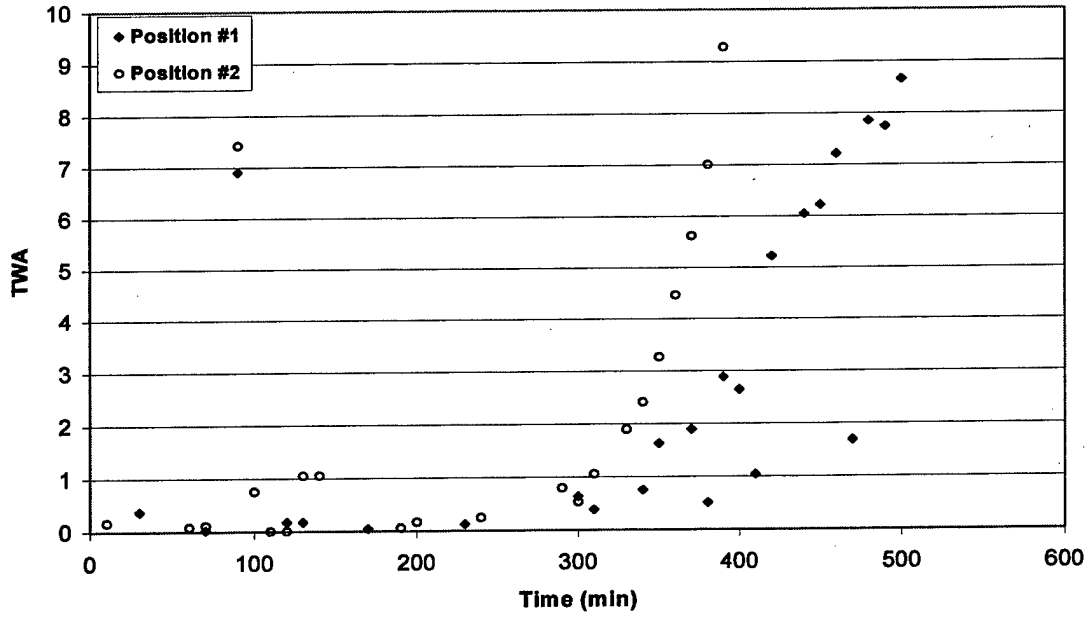


Figure A.2. Breakthrough Data for the 01/28/03 Experiment
Vapure 612, 3cm, 73°C, 12cm/s, Dry

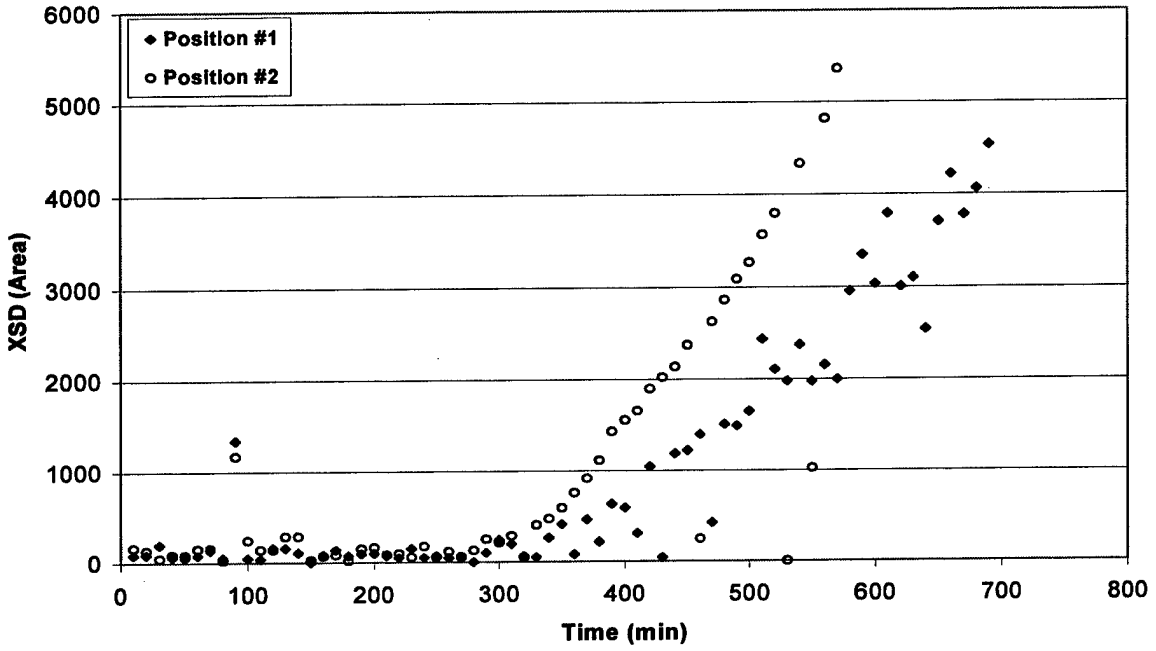


Figure A.3. Breakthrough Data for the 01/29/03 Experiment
Vapure 612, 3.5cm, 73°C, 12cm/s, Dry

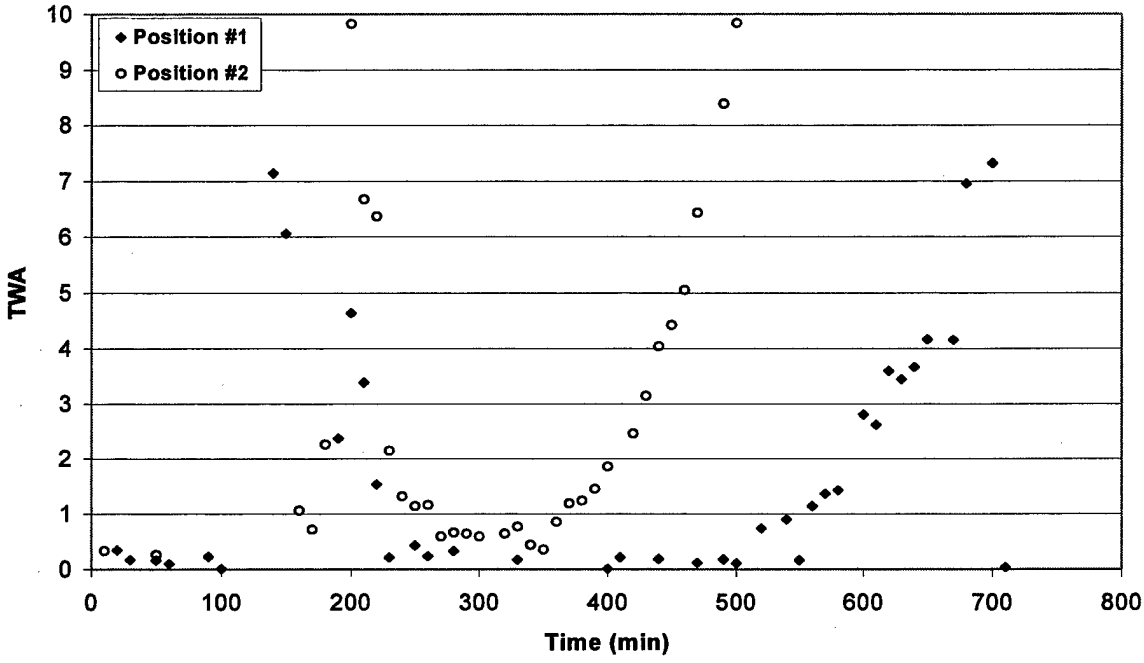


Figure A.4. Breakthrough Data for the 01/29/03 Experiment
Vapure 612, 3.5cm, 73°C, 12cm/s, Dry

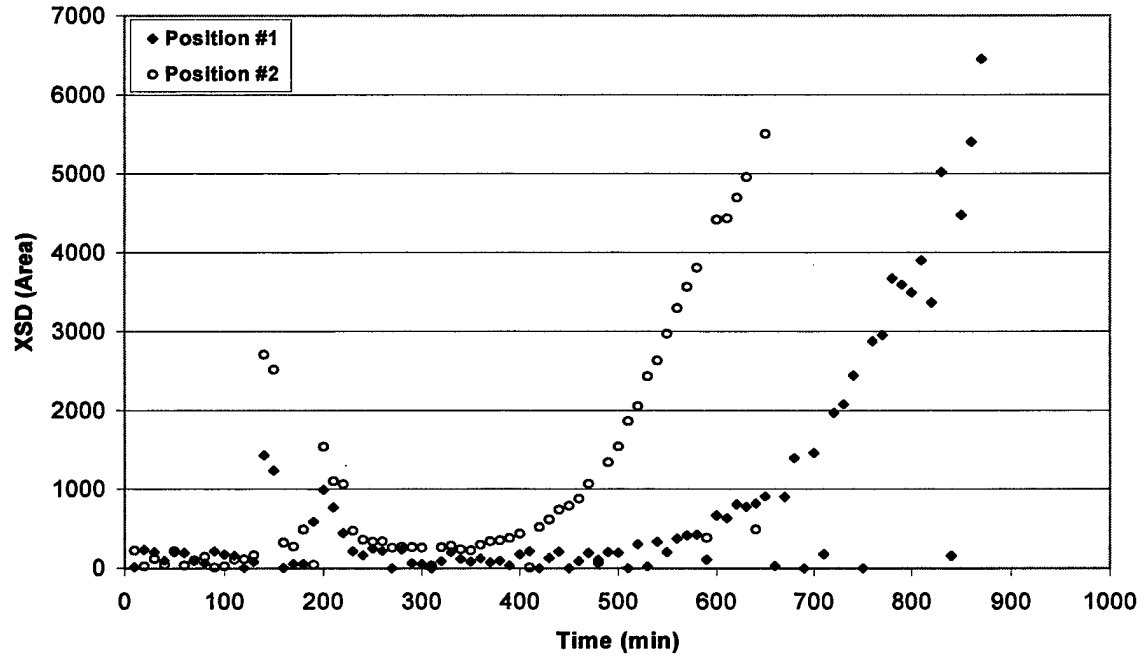


Figure A.5. Breakthrough Data for the 01/31/03 Experiment
Vapure 612, 4cm, 73°C, 12cm/s, Dry

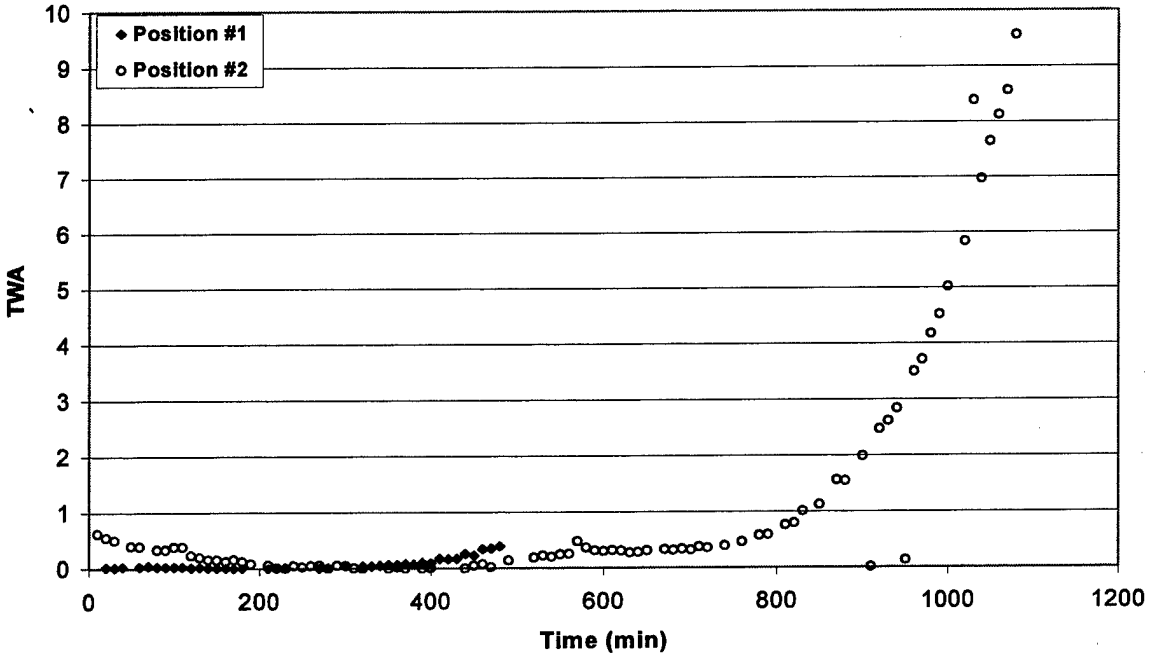


Figure A.6. Breakthrough Data for the 01/31/03 Experiment
Vapure 612, 4cm, 73°C, 12cm/s, Dry

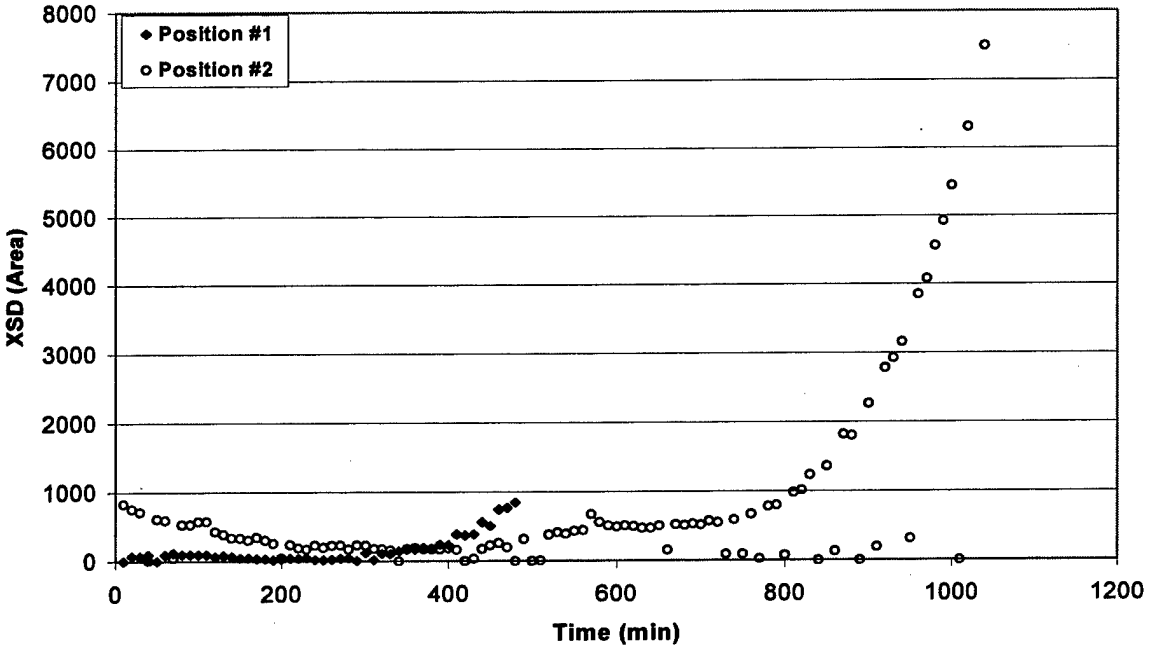


Figure A.7. Breakthrough Data for the 02/05/03 Experiment
Vapure 612, 2.5cm, 73°C, 12cm/s, Dry

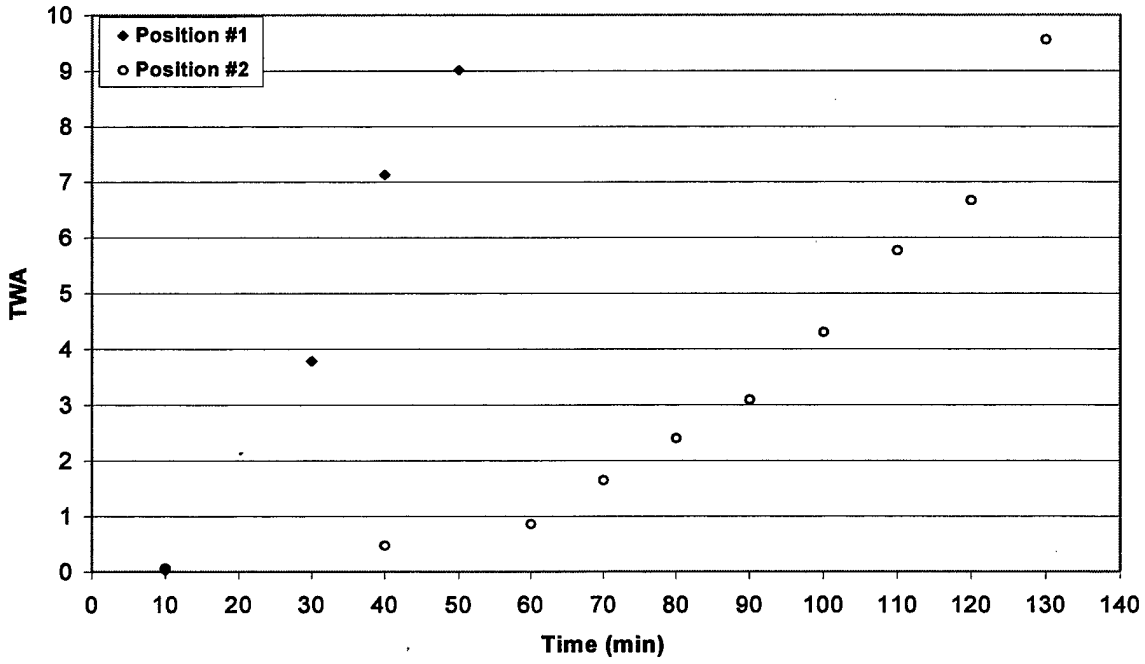


Figure A.8. Breakthrough Data for the 02/05/03 Experiment
Vapure 612, 2.5cm, 73°C, 12cm/s, Dry

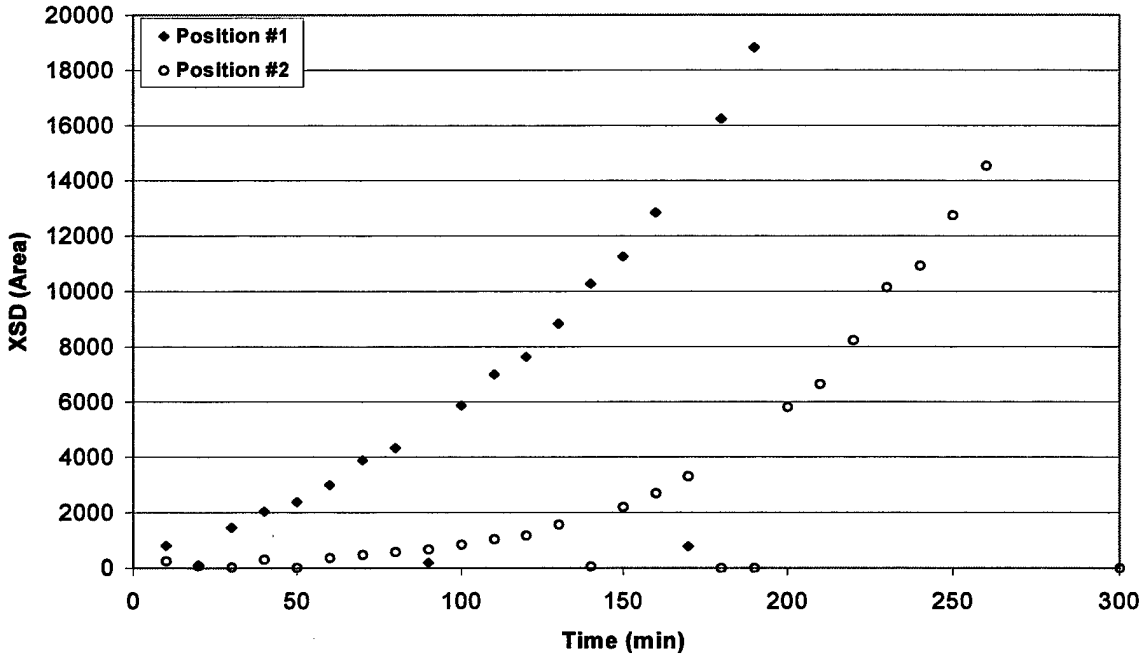


Figure A.9. Breakthrough Data for the 02/06/03 Experiment
Vapure 612, 4cm, 73°C, 12cm/s, Dry

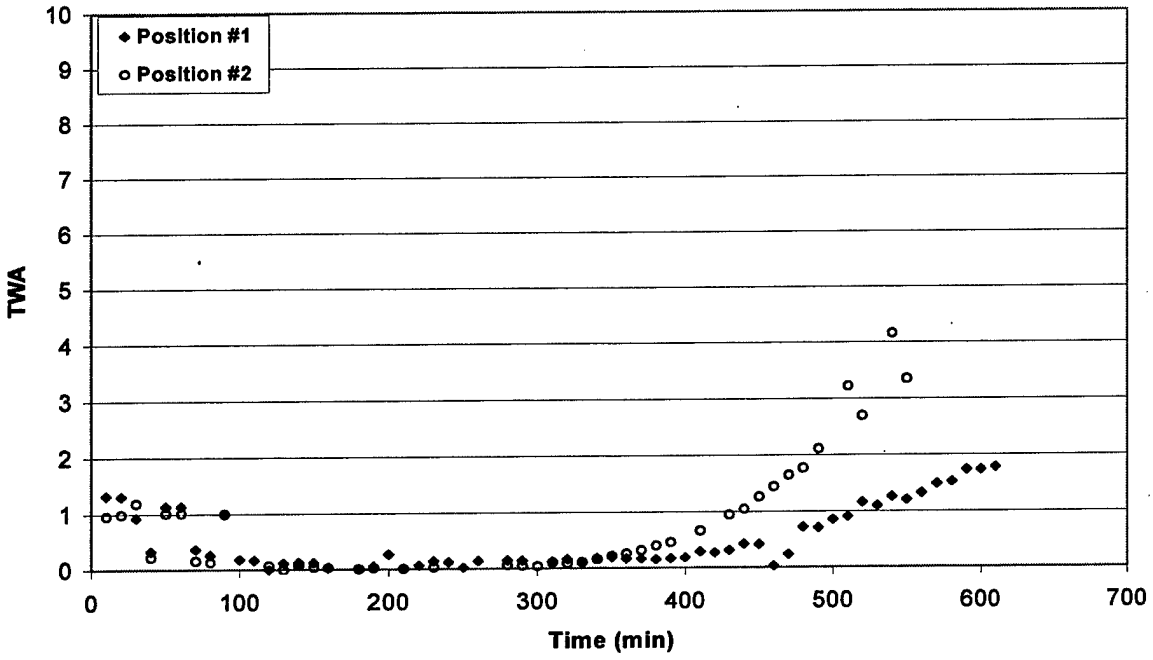


Figure A.10. Breakthrough Data for the 02/06/03 Experiment
Vapure 612, 4cm, 73°C, 12cm/s, Dry

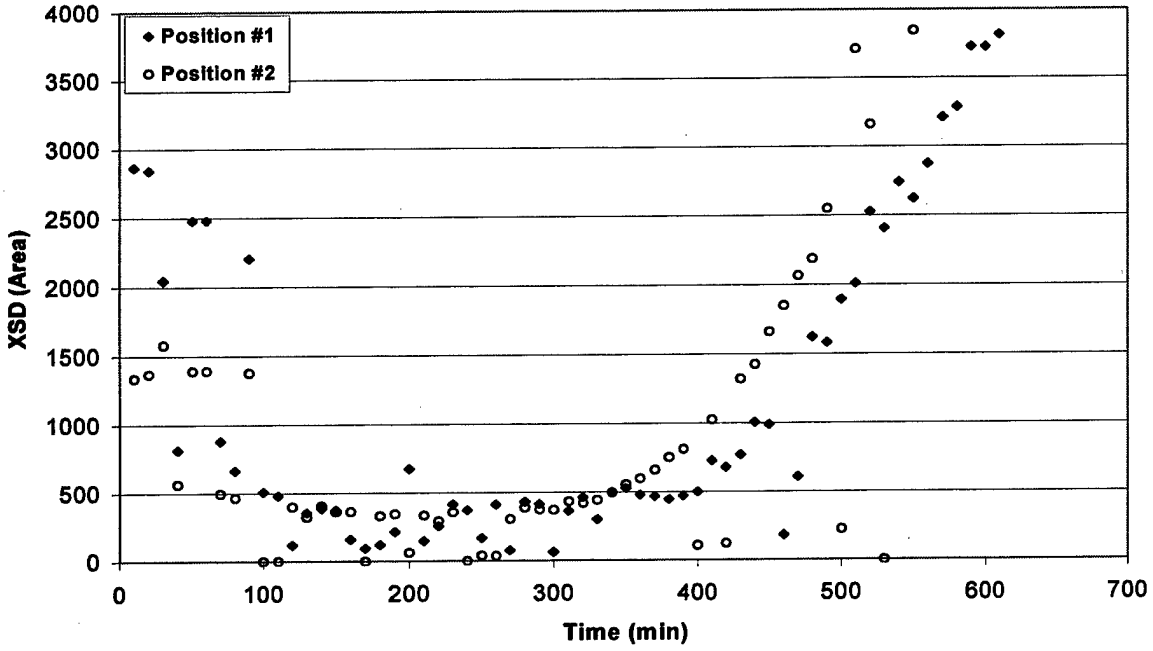


Figure A.11. Breakthrough Data for the 02/13/03 Experiment
Vapure 612, 3cm, 73°C, 12cm/s, Dry

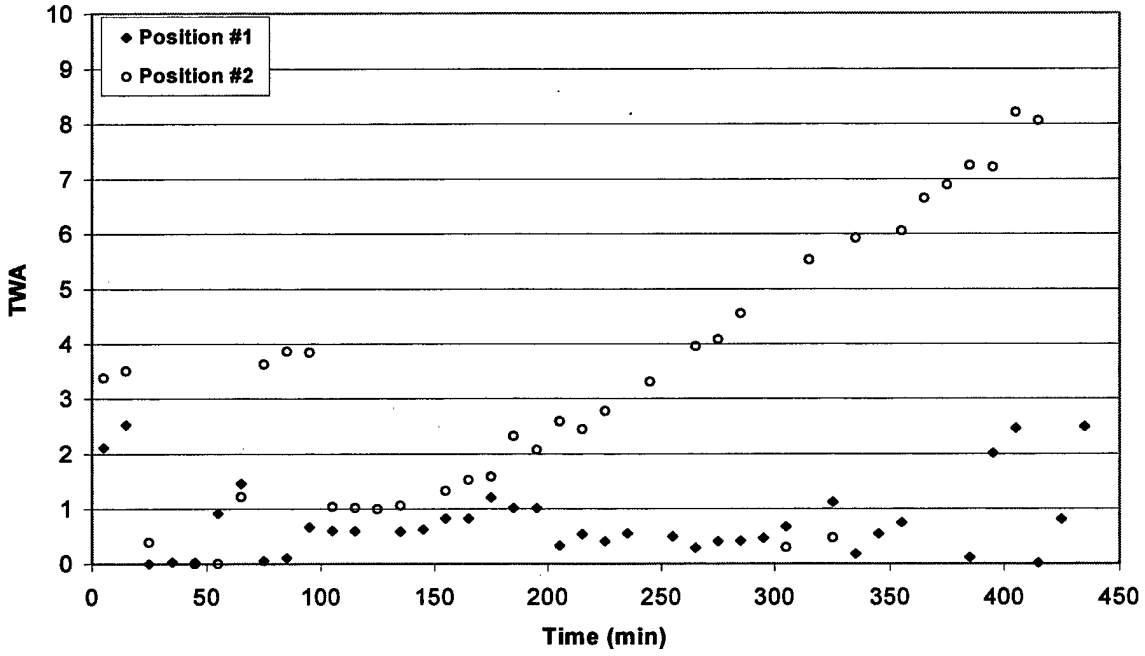


Figure A.12. Breakthrough Data for the 02/13/03 Experiment
Vapure 612, 3cm, 73°C, 12cm/s, Dry

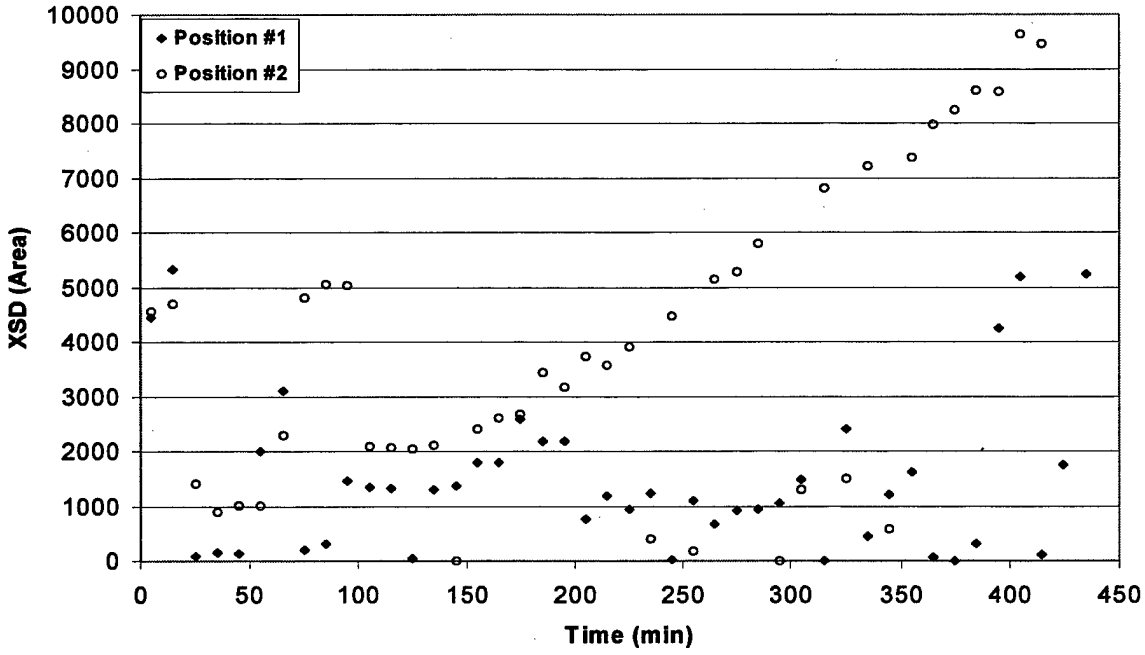


Figure A.13. Breakthrough Data for the 02/14/03 Experiment
Vapure 612, 3cm, 73°C, 12cm/s, Dry

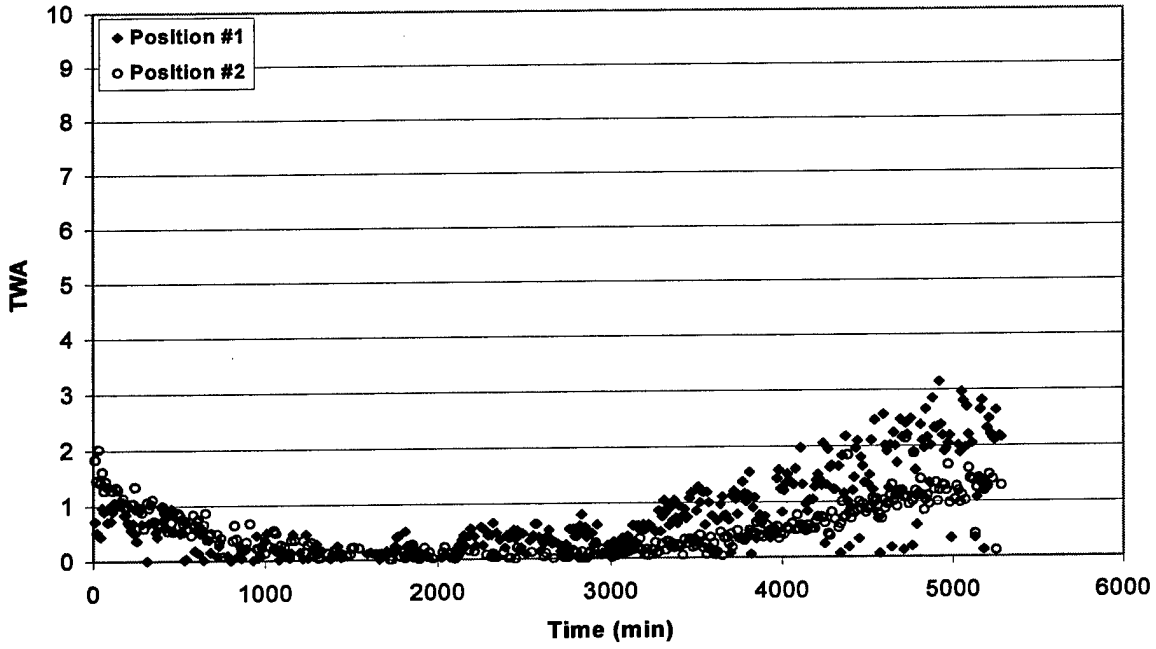


Figure A.14. Breakthrough Data for the 02/14/03 Experiment
Vapure 612, 3cm, 73°C, 12cm/s, Dry

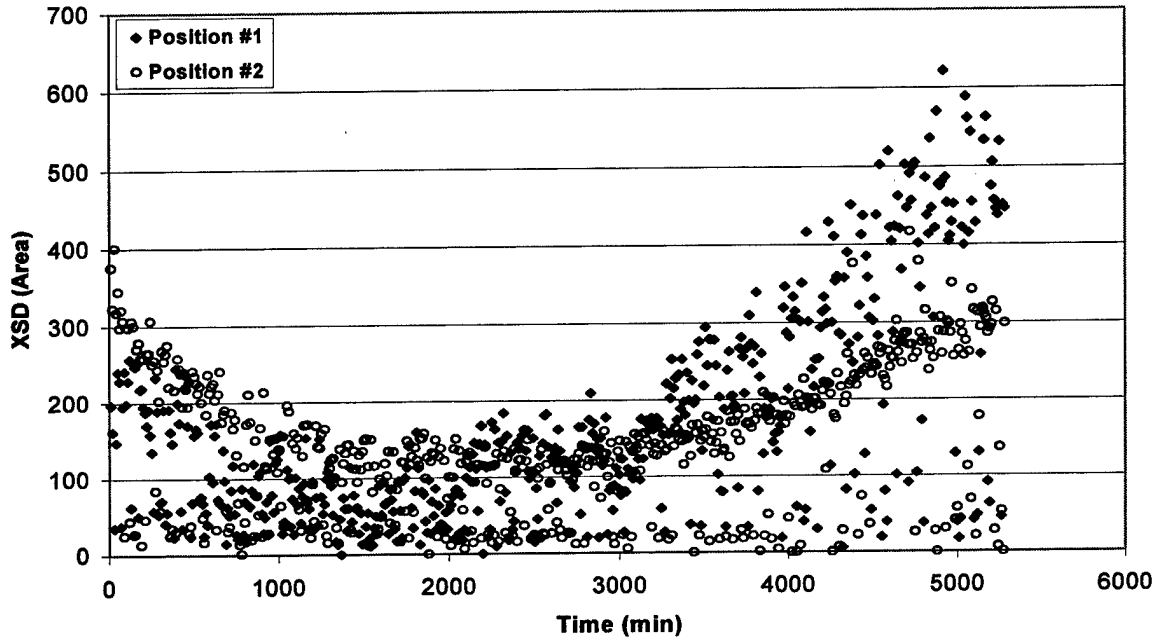


Figure A.15. Breakthrough Data for the 02/20/03 Experiment
Vapure 612, 2.5cm, 73°C, 12cm/s, Dry

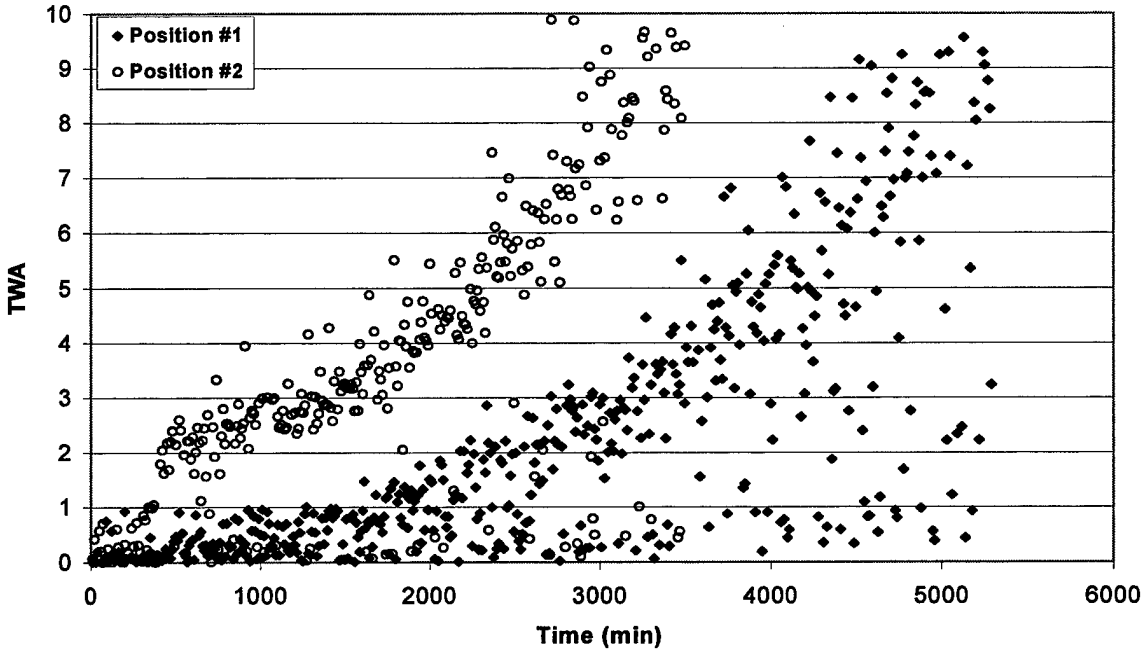


Figure A.16. Breakthrough Data for the 02/20/03 Experiment
Vapure 612, 2.5cm, 73°C, 12cm/s, Dry

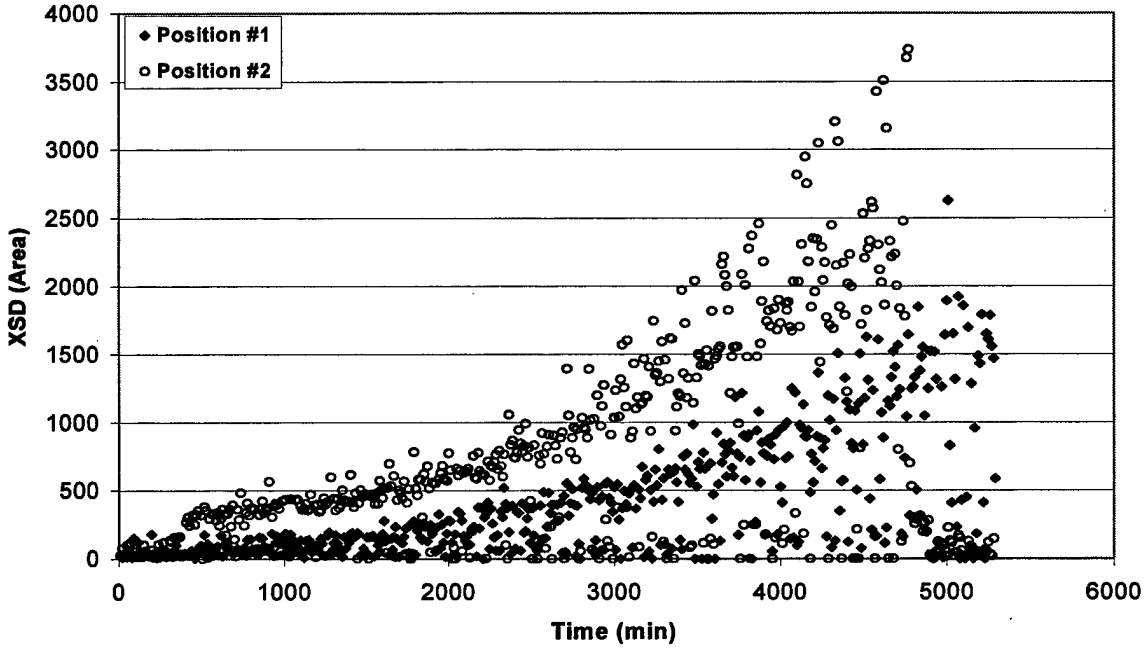


Figure A.17. Breakthrough Data for the 02/25/03 Experiment
ASZM-T 12x30 Mesh, 2cm, 6cm/s, 30°C, Dry

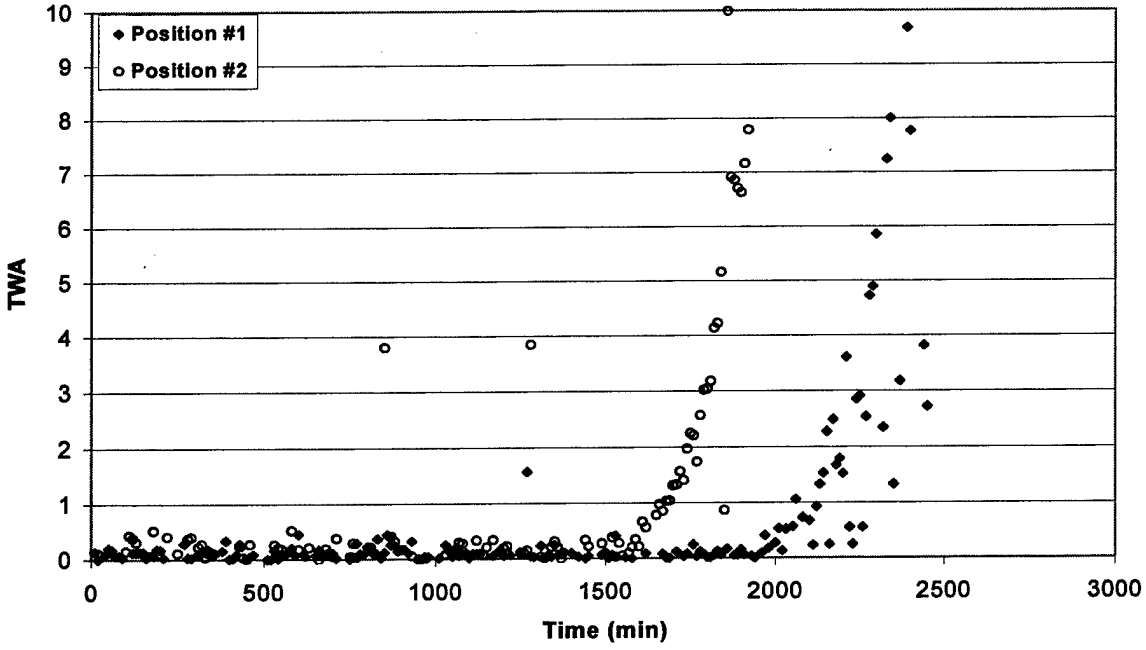


Figure A.18. Breakthrough Data for the 02/25/03 Experiment
ASZM-T 12x30 Mesh, 2cm, 6cm/s, 30°C Dry

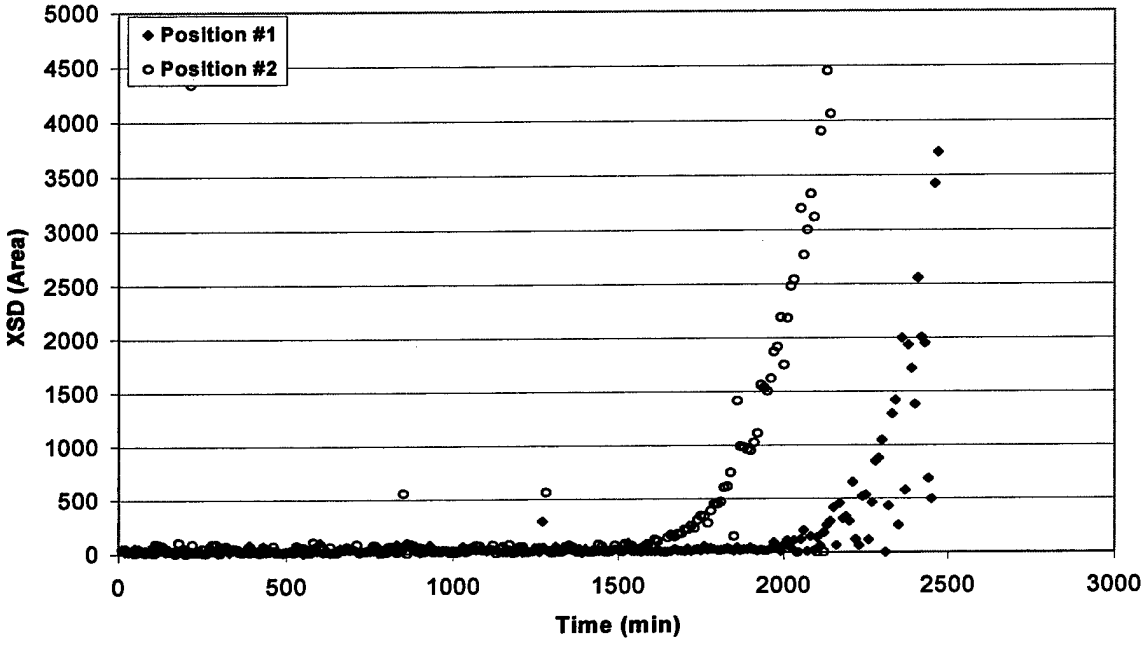


Figure A.19. Breakthrough Data for the 02/28/03 Experiment
Vapure 612, 2.5cm, 45°C, 12cm/s, Dry

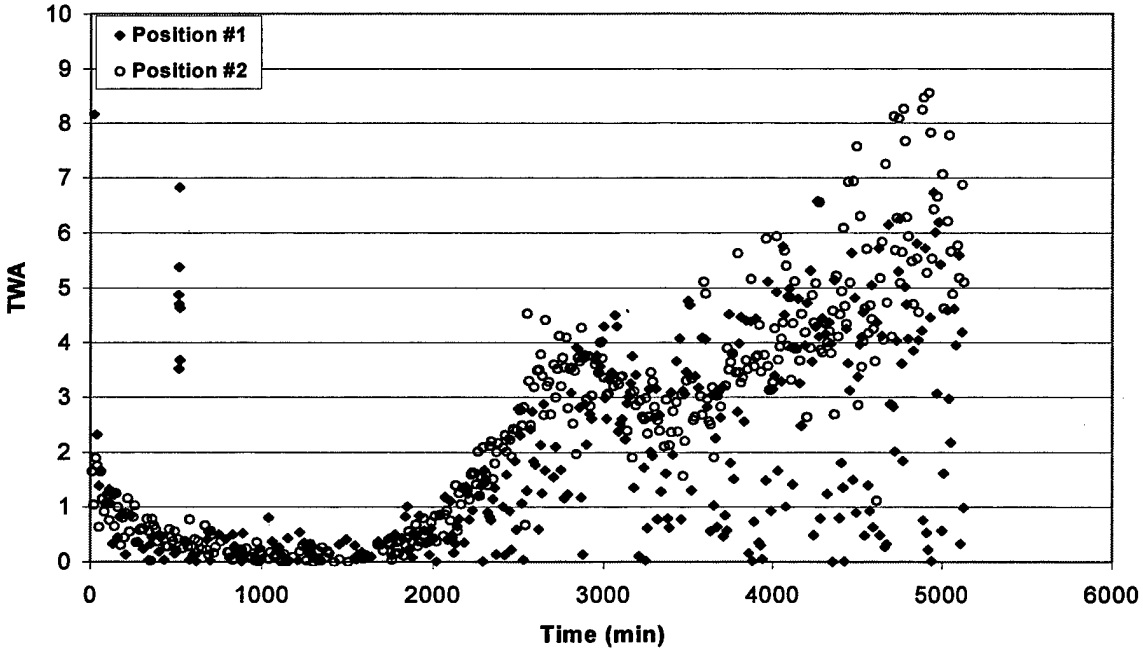


Figure A.20. Breakthrough Data for the 02/28/03 Experiment
Vapure 612, 2.5cm, 45°C, 12cm/s, Dry

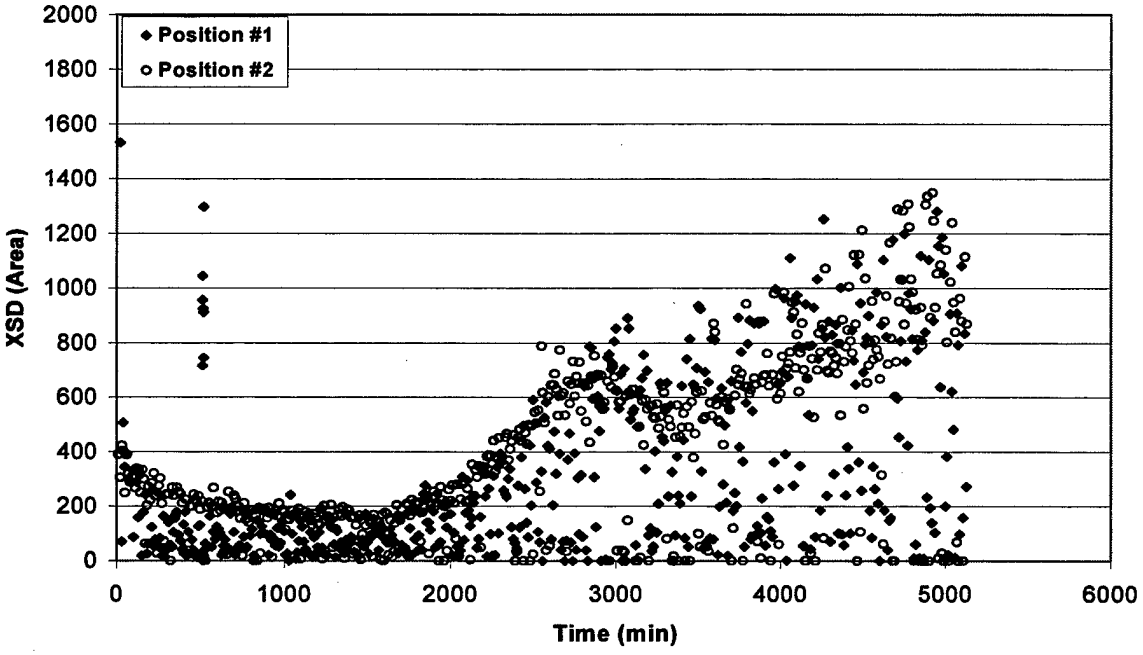


Figure A.21. Breakthrough Data for the 03/05/03 Experiment
ASZM-T 12x30 Mesh, 2cm, 6cm/s, 30°C, Dry

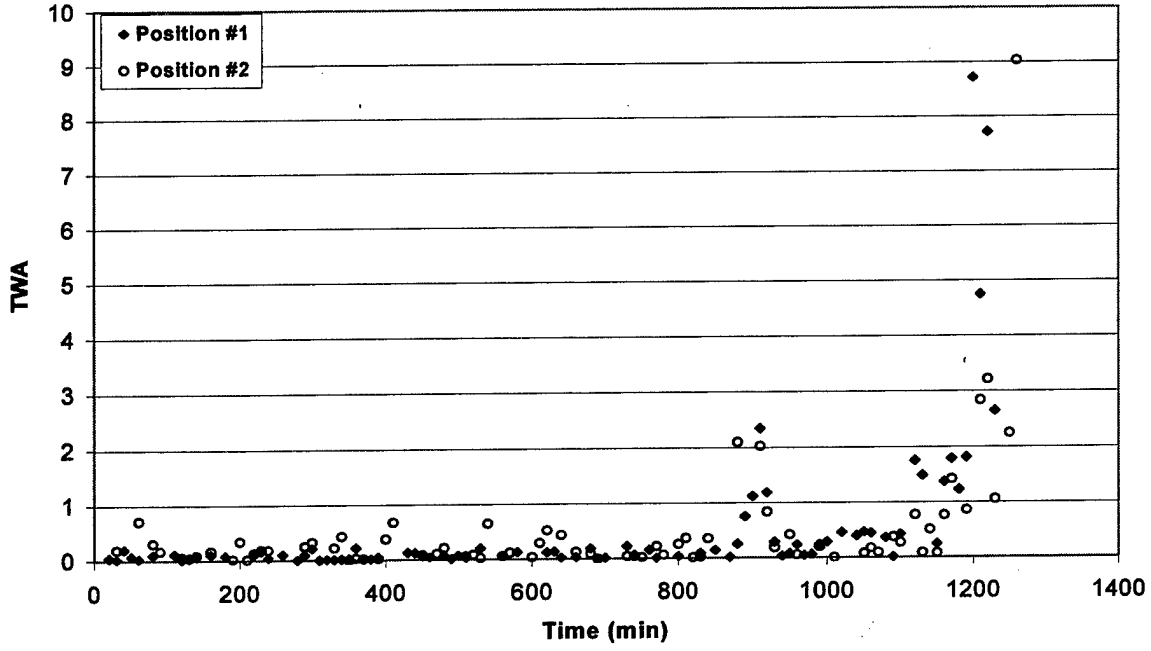


Figure A.22. Breakthrough Data for the 03/05/03 Experiment
ASZM-T 12x30 Mesh, 2cm, 6cm/s, 30°C, Dry

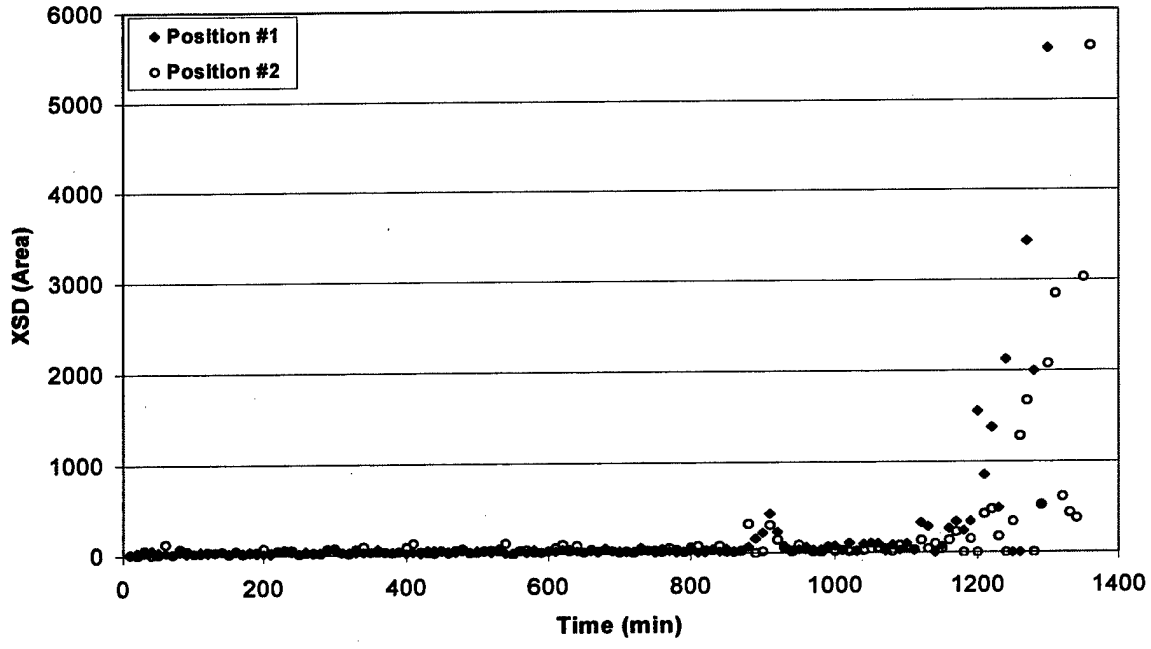


Figure A.23. Breakthrough Data for the 03/13/03 Experiment Vapure 612, 2.5cm, 12cm/s, 45°C, 25%RH

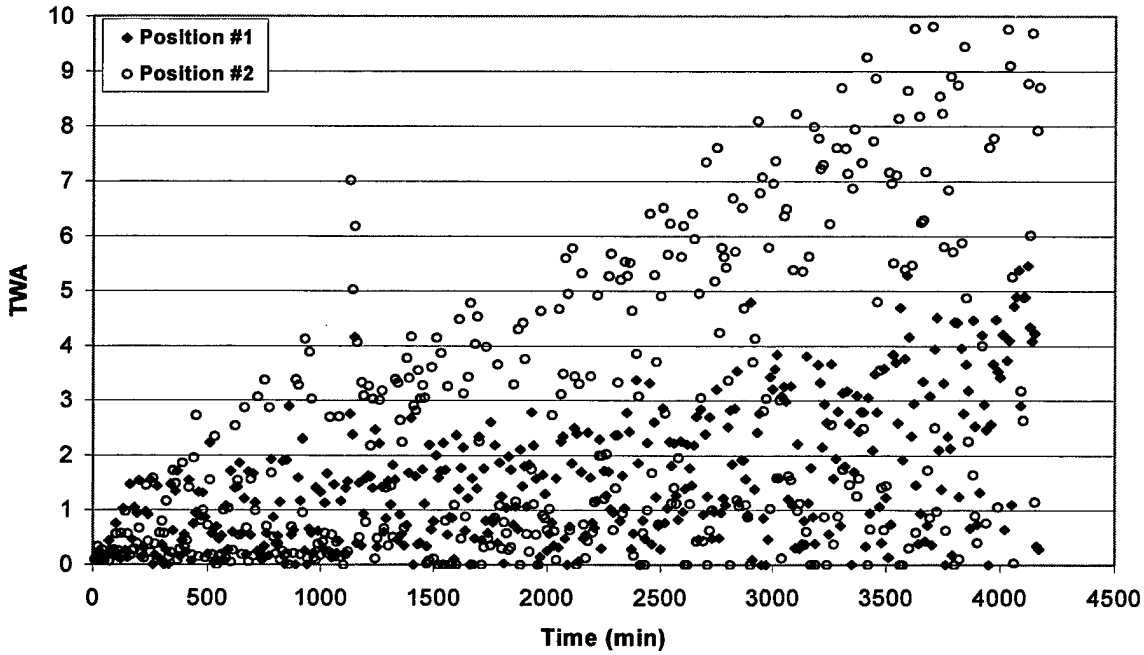


Figure A.24. Breakthrough Data for the 03/13/03 Experiment Vapure 612, 2.5cm, 12cm/s, 45°C, 25%RH

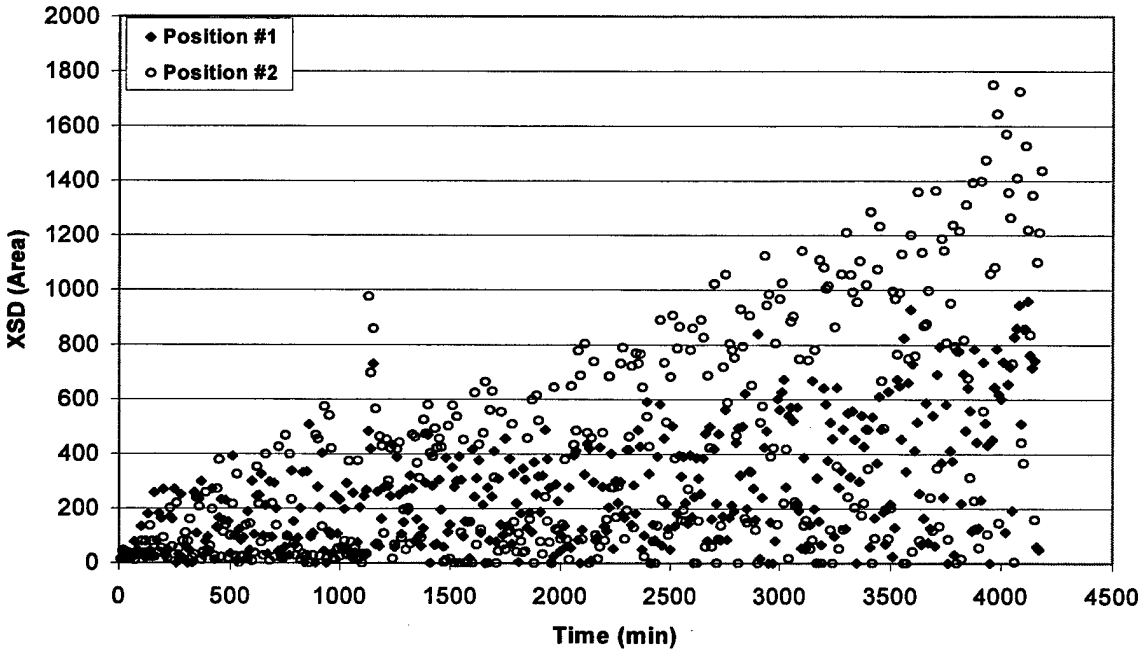


Figure A.25. Breakthrough Data for the 03/24/03 Experiment
ASZM-T 12x30 Mesh, 2cm, 6cm/s, 30°C, 40%RH

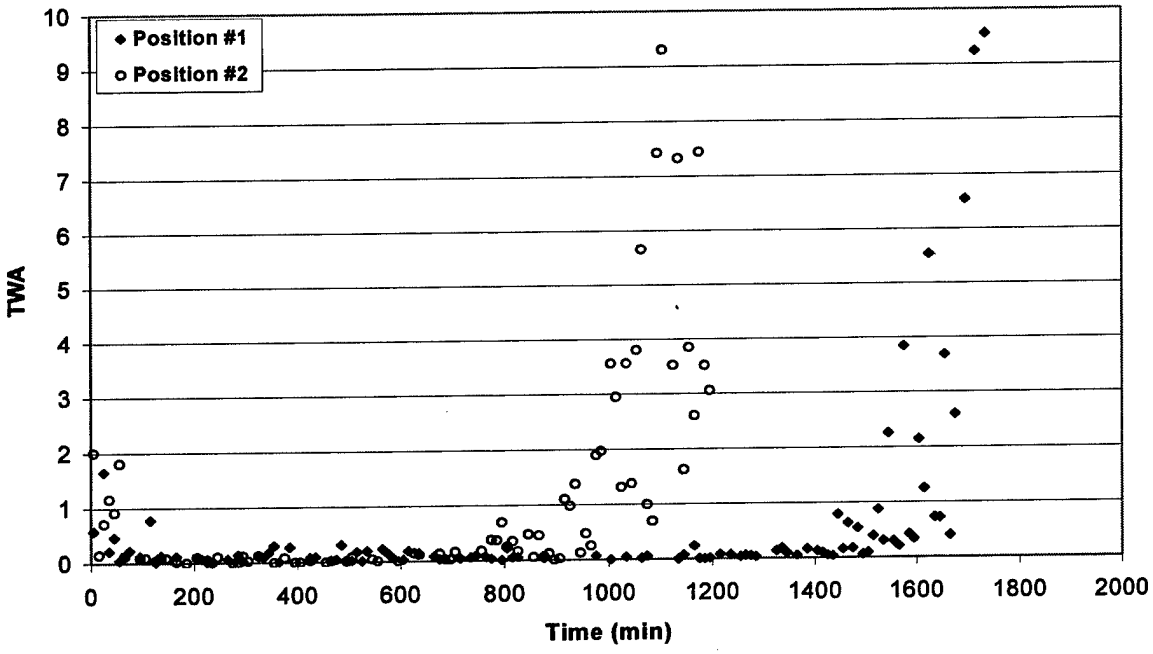


Figure A.26. Breakthrough Data for the 03/24/03 Experiment
ASZM-T 12x30 Mesh, 2cm, 6cm/s, 30°C, 40%RH

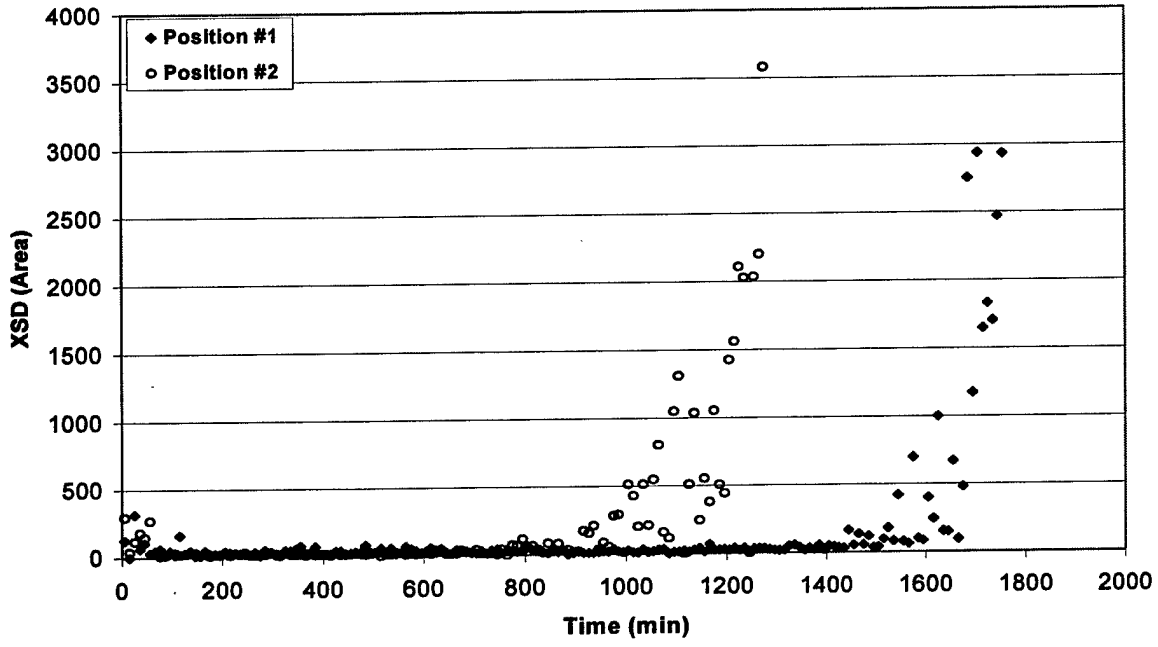


Figure A.27. Breakthrough Data for the 03/28/03 Experiment ASZM-T 12x30 Mesh, 2cm, 6cm/s, 30°C, 40%RH

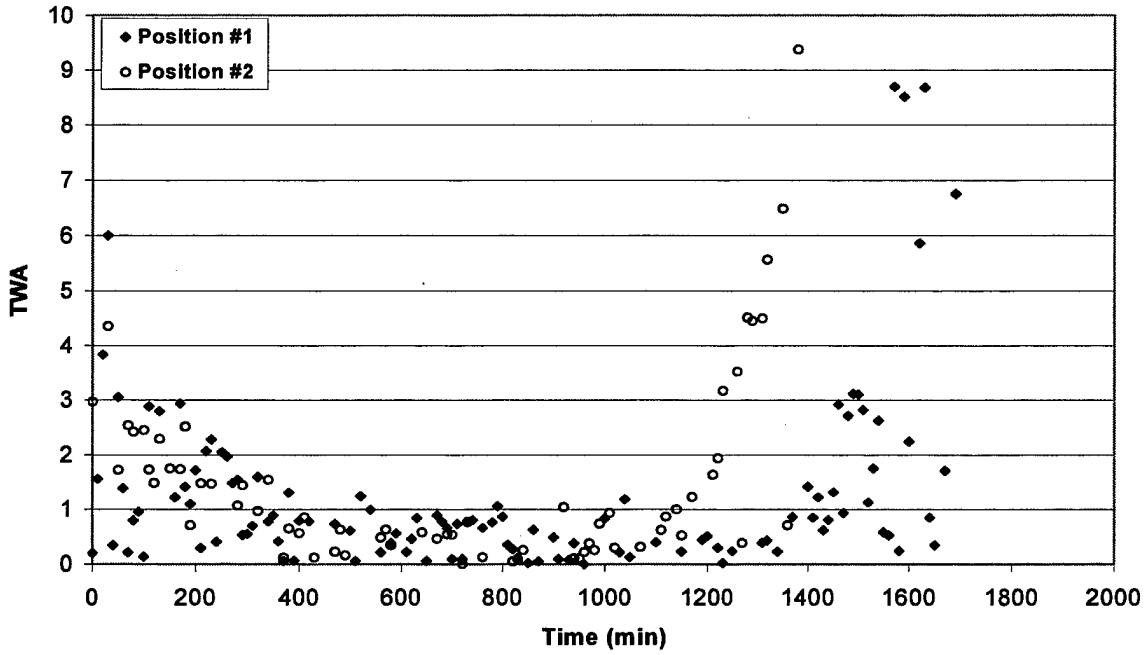


Figure A.28. Breakthrough Data for the 03/28/03 Experiment ASZM-T 12x30 Mesh, 2cm, 6cm/s, 30°C, 40%RH

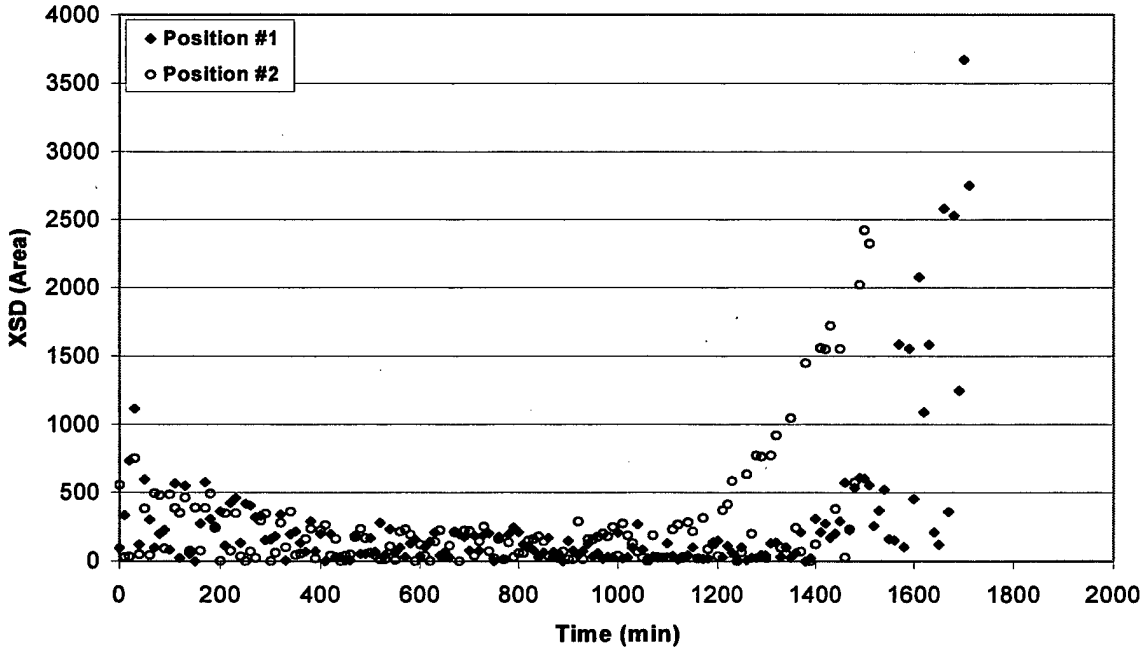


Figure A.29. Breakthrough Data for the 04/01/03 Experiment
Vapure 612, 5cm, 12cm/s, 45°C, 25%RH

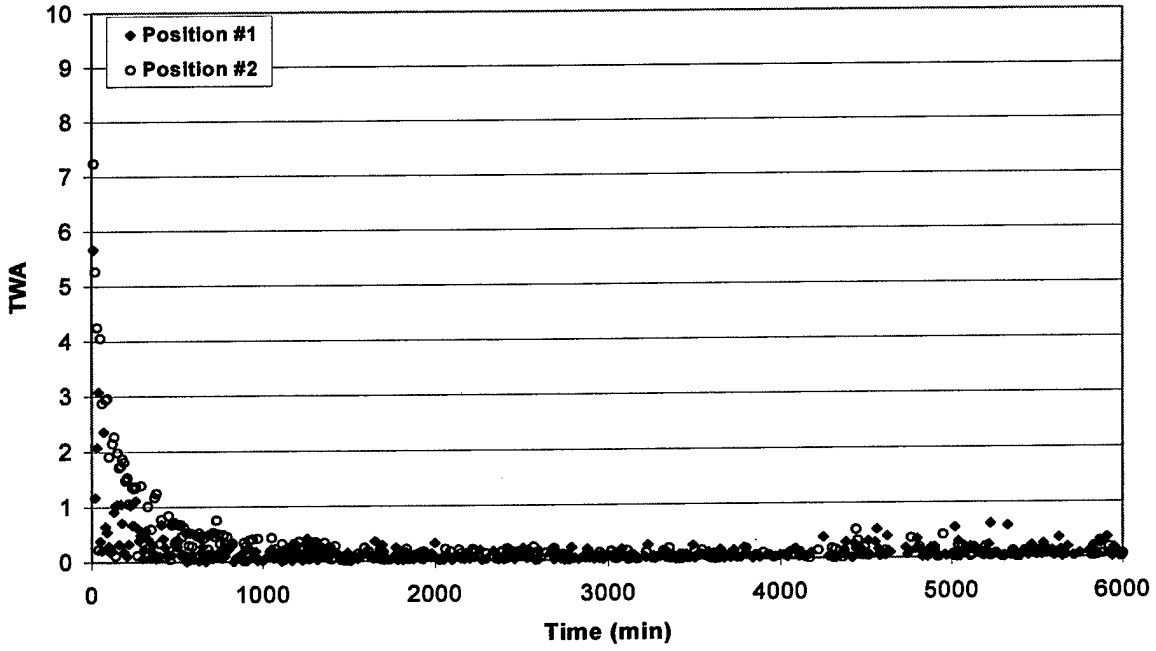


Figure A.30. Breakthrough Data for the 04/01/03 Experiment
Vapure 612, 5cm, 12cm/s, 45°C, 25%RH

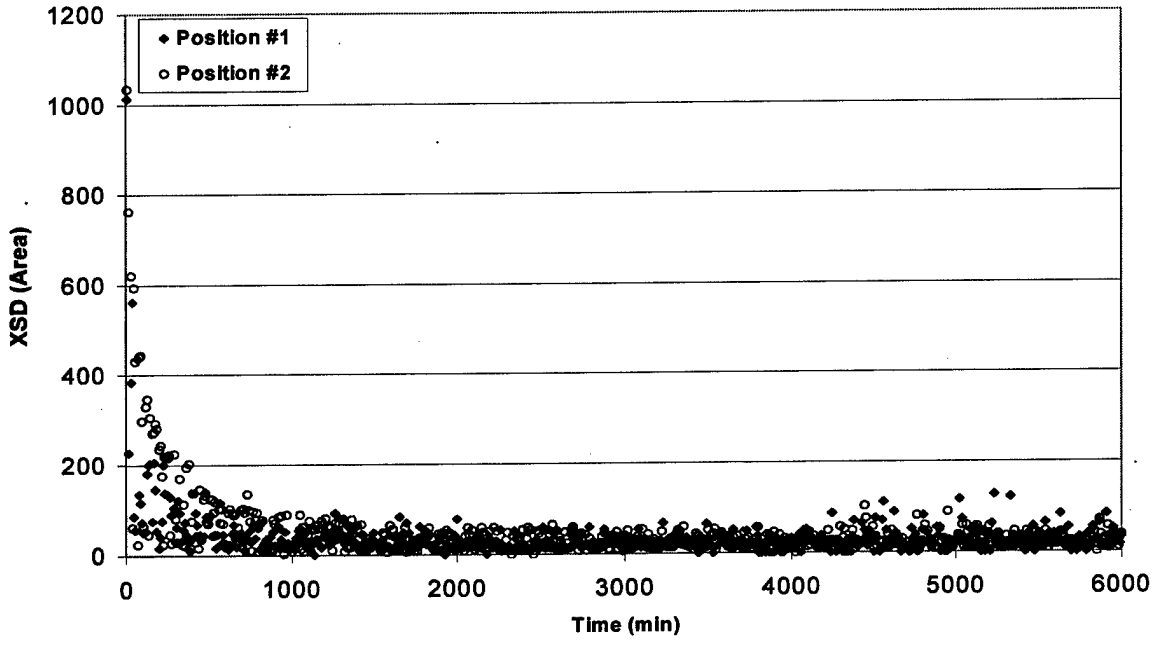


Figure A.31. Breakthrough Data for the 04/11/03 Experiment
ASZM-T 6x16 Mesh, 4cm, 12cm/s, 73°C, Dry

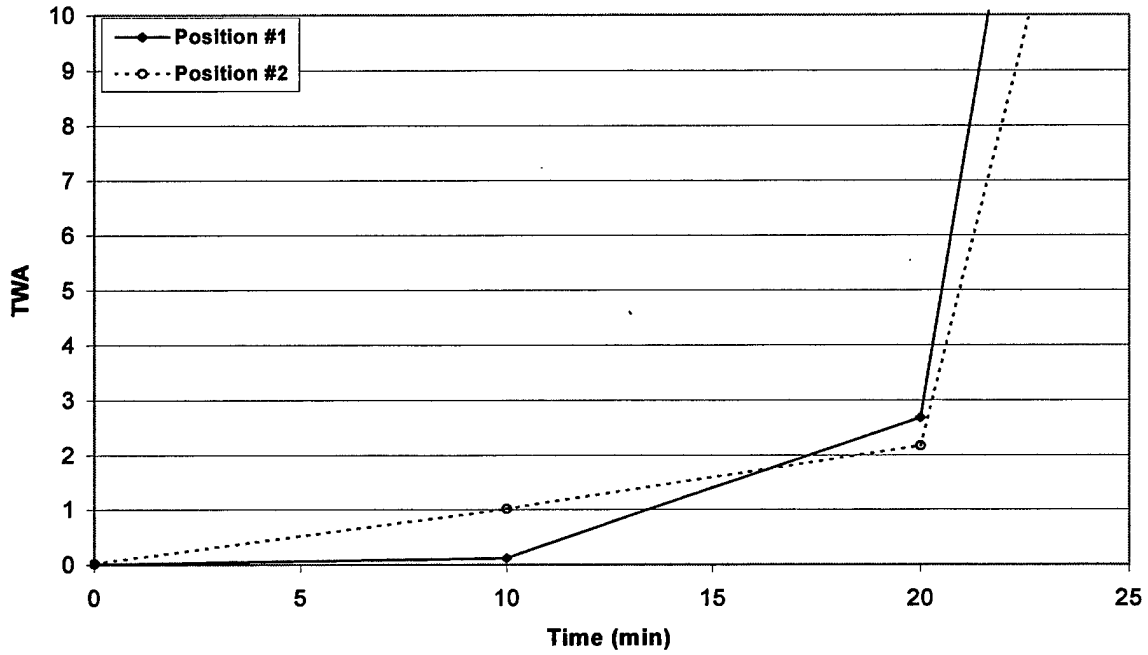


Figure A.32. Breakthrough Data for the 04/11/03 Experiment
ASZM-T 6x16 Mesh, 4cm, 12cm/s, 73°C, Dry

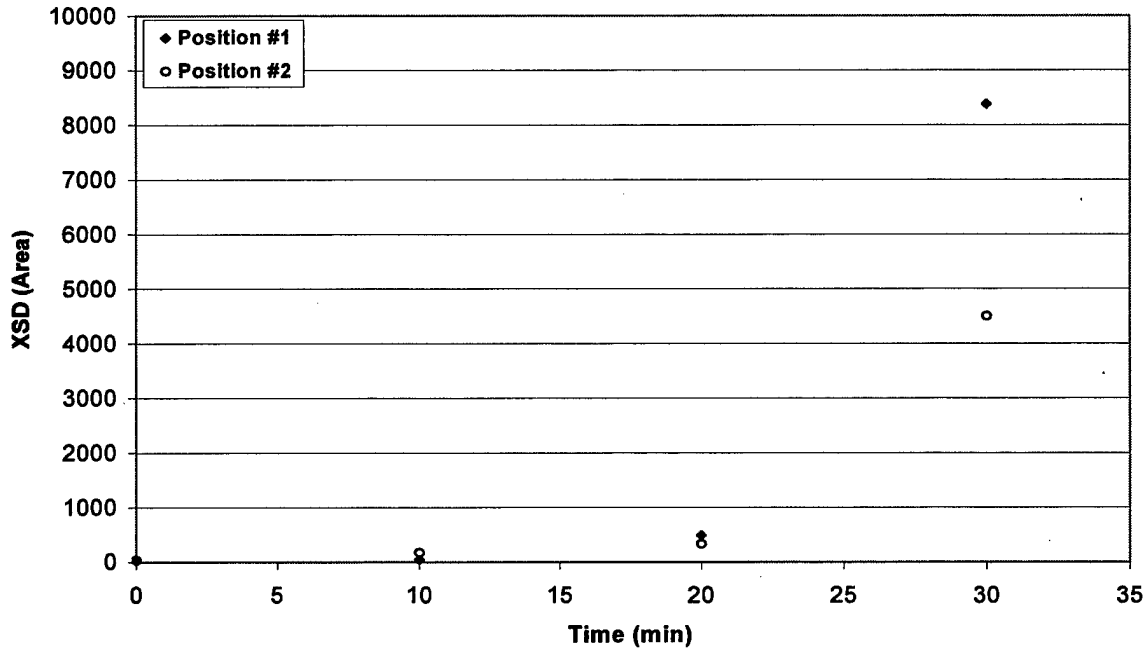


Figure A.33. Breakthrough Data for the 04/14/03 Experiment ASZM-T 6x16 Mesh, 4cm, 12cm/s, 45°C, 25%RH

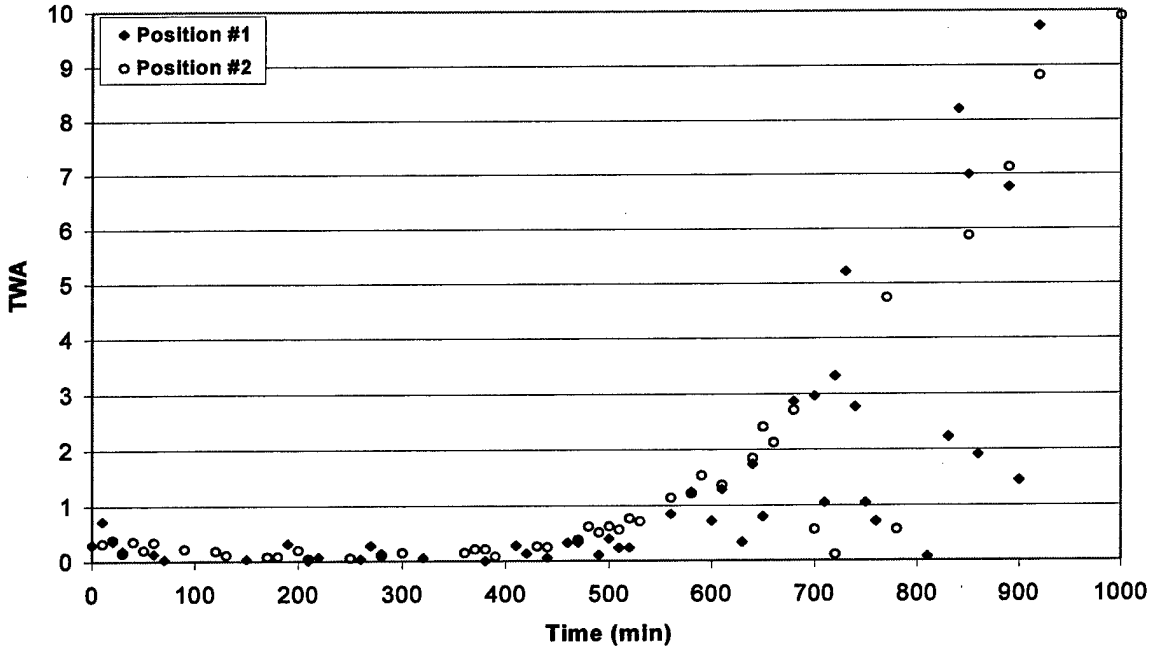


Figure A.34. Breakthrough Data for the 04/14/03 Experiment ASZM-T 6x16 Mesh, 4cm, 12cm/s, 45°C, 25%RH

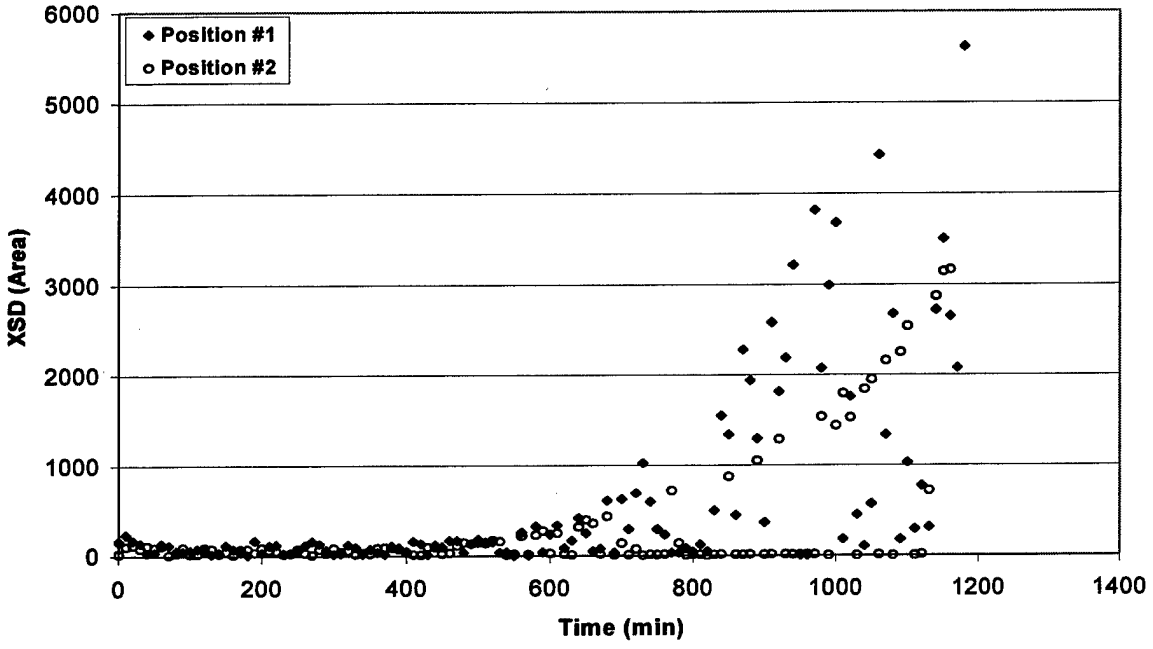


Figure A.35. Breakthrough Data for the 04/16/03 Experiment
ASZM-T 6x16 Mesh, 4cm, 12cm/s, 45°C, 25%RH

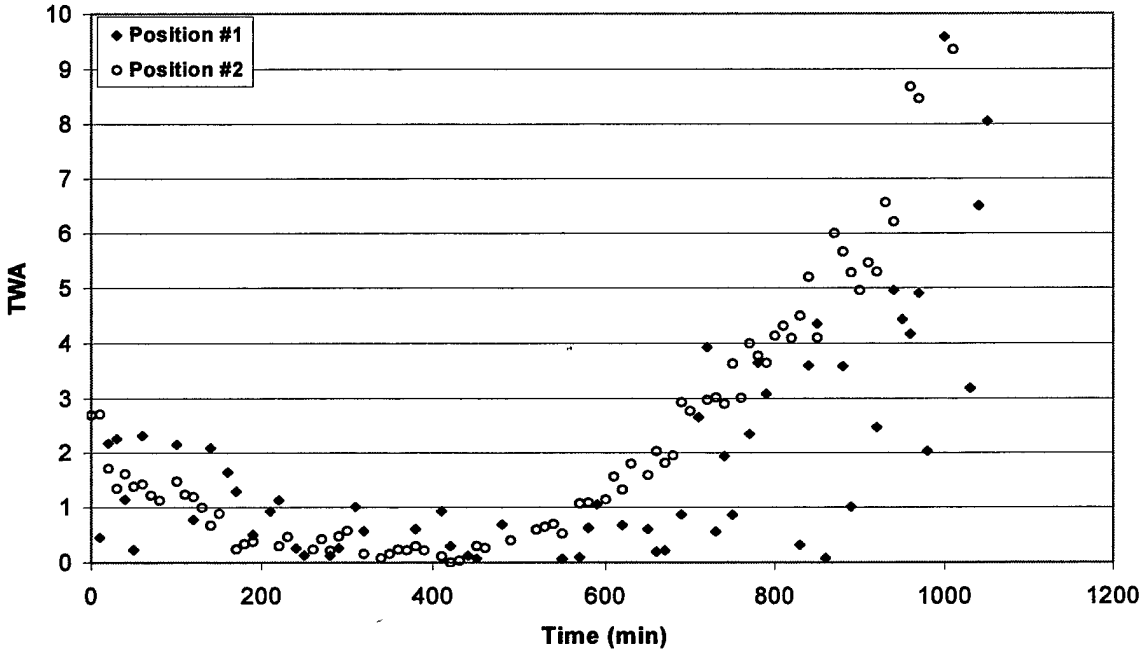
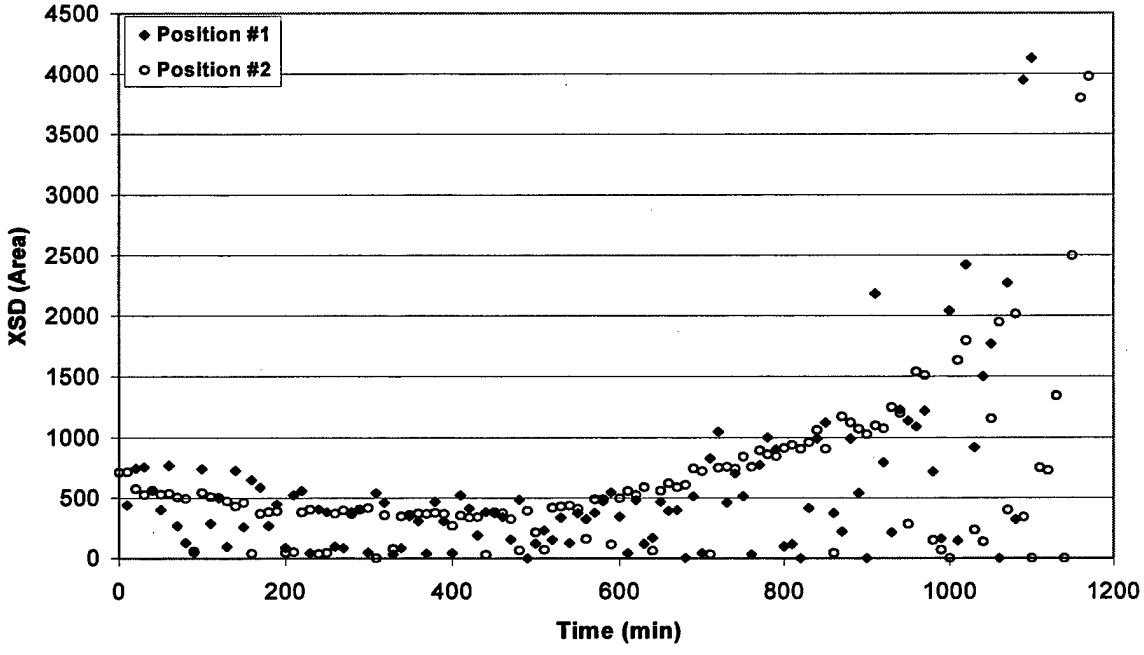


Figure A.36. Breakthrough Data for the 04/16/03 Experiment
ASZM-T 6x16 Mesh, 4cm, 12cm/s, 45°C, 25%RH



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APPENDIX B

MINICAMS OPERATING PARAMETERS

		BED 1			BED 2			BOX		
TEMPERATURES, °C:	Ambient	S 40	a 40	± 15	s 40	a 40	± 15	s 40	a 40	± 15
	Inlet	S100	a100	± 15	s100	a100	± 15	s100	a100	± 15
	DET	S150	a150	± 15	s150	a150	± 15	s150	a150	± 15
	FLA	S 0	a 43	± 99	s 0	a 43	± 99	s 0	a 43	± 99
	Column, low	S 50	a 50	± 15	s 50	a 50	± 15	s 70	a 50	± 15
	Column, high	S190	a190	± 15	s190	a190	± 15	s200	a190	± 15
	PCT heater, low	S 50	a 50	± 20	s 50	a 50	± 20	s 50	a 50	± 20
	PCT heater, high	s250	a250	±30	s250	a250	±30	s250	a250	±30
PRESSURES, psi:	Hydrogen	s 15	a 15	± 5	s 15	a 15	± 5	s 15	a 15	± 5
	Air	s 15	a 15	± 5	s 15	a 15	± 5	s 15	a 15	± 5
	Nitrogen	s 25	a 25	± 5	s 25	a 25	± 5	s 25	a 25	± 5
SAMPLE:	Flow rate, sccm	s400	a400	± 20	s400	a400	± 20	s400	a400	± 20
EDT	Flow rate, sccm	s 15	a 15	± 1	a 15	a 15	± 1	a 15	a 15	± 1
TIMES, sec:	Purge	b 0	e300		b 0	e300		b 0	e240	
	Desorb	b 20	e 70		b 20	e 70		b 20	e 70	
	Column purge	b 70	e285		b 70	e285		b 70	e225	
	Sample	b300	e600		b300	e600		b240	e600	
	Inject	b300	e310		b300	e310		b240	e250	
	COLF	b285			b285			b225		
	PKW	±8			±8			±4		
XSD TEMPERATURE		900 °C			900 °C			900 °C		
AGENT GATE		b246	e269		b265	e289		b123	e137	
CONCENTRATION REPORTS:	Peak Area									